

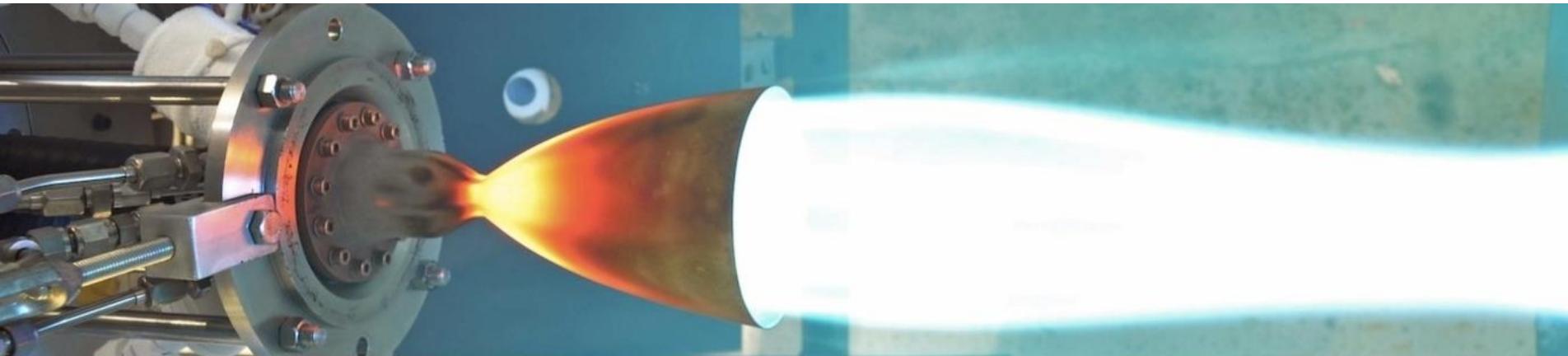


GRC Webinar – Additive Manufacturing High Temperature Alloys

Dr. Tim Smith PhD

LEW-20020-1: “Novel Fabrication Technique for Oxide Dispersion Strengthened (ODS) Alloys”

LEW-19886-1: “Additively Manufactured Oxide Dispersion Strengthened Medium Entropy Alloys for High Temperature Applications”





Tim Smith - Bio



2011: Graduated from Wright State University with a Bachelors degree in Mechanical Engineering

2016: Graduated from Ohio State University with a PhD in Materials Science

2015: Began career at the NASA Glenn Research center through the Pathways Internship Program

2016-Present: Currently works at GRC where his research focuses on the production, microstructural characterization, and deformation analysis of high temperature alloys.

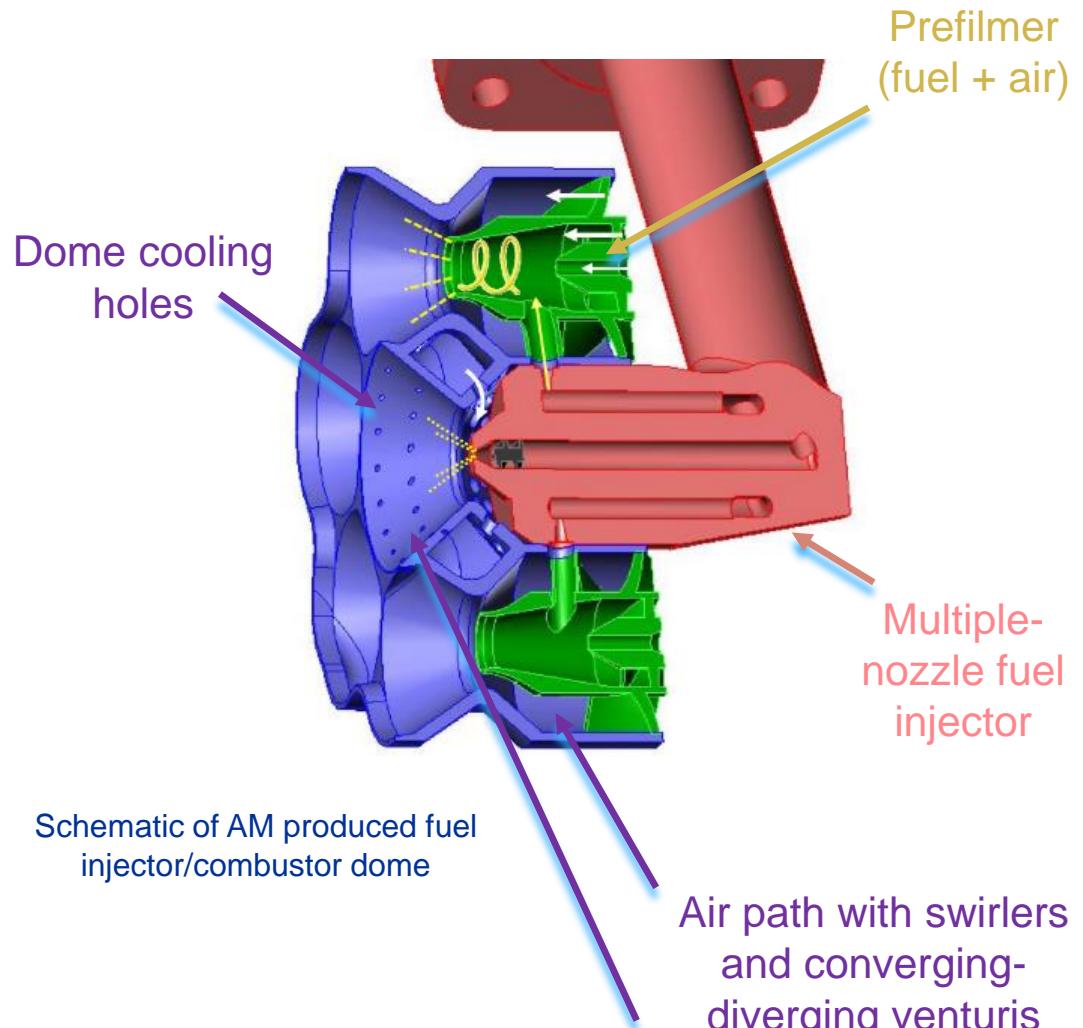
He has contributed to 29 peer-reviewed journal articles including publications in Nature Communications and Nature Communications Materials.

Background – NASA Application

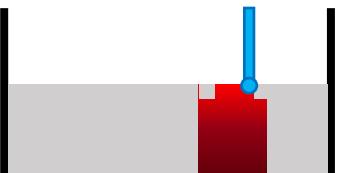
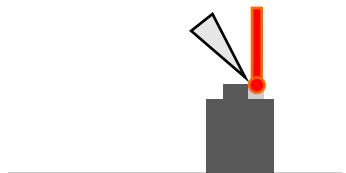
Problem: Conventional materials and processing techniques limit the design of combustor domes used in jet turbine engines.

Proposed Solution: Develop a high ductility, high temperature material for an additively-manufactured (AM) combustor fuel nozzle and dome for supersonic aircraft ($>1093^{\circ}\text{C}$ (2000°F) operating temperature).

- Lead to several improvements to the turbine combustor design ultimately reducing NOx pollution and lowering weight.
- May enable lean-front-end small-core combustors.



Metallic Additive Manufacturing

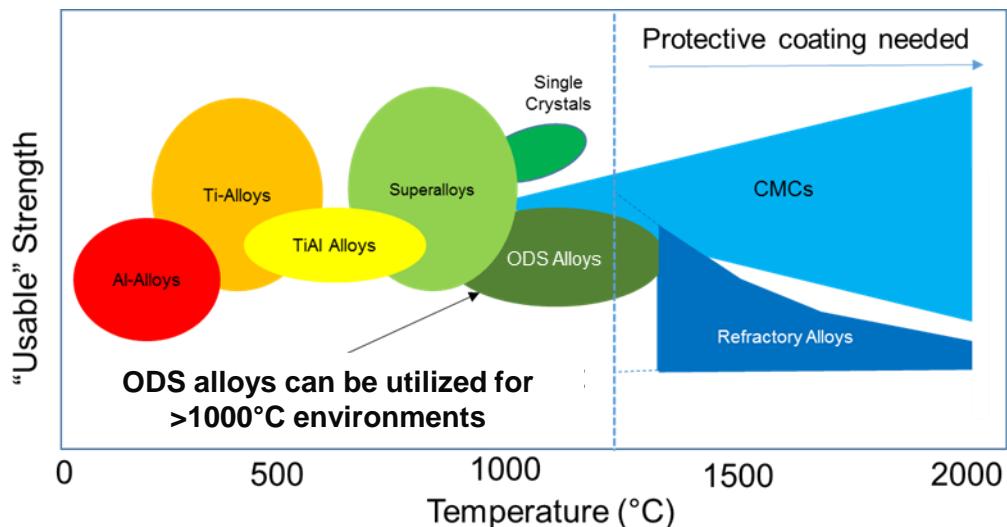
Process	Laser Powder Bed Fusion (L-PBF)	Electron Beam Powder Bed Fusion	Direct Energy Deposition (DED)
			
Energy Source	Laser	E-Beam	Laser or E-Beam
Powder Bed	Yes	Yes	No
Power (W or kV)	50-1000 W	30-60kV	100-2000 W
Max Build Size (mm)	500 x 280 x 320	500 x 280 x 320	2000 x 1500 x 750
Material	Metallic Powder	Metallic Powder	Metallic Powder or Wire
Dimensional Accuracy	<0.04 mm	0.04-0.2 mm	0.5 mm (powder) 1.0 mm (wire)

- 3D printing or additive manufacturing (AM) has shown promise in realizing a new design space for aerospace applications.
- Each AM technique has a set of pros and cons associated with them.
- Instead of producing well known cast and wrought alloys with AM. We should look at AM as a new opportunity to produce materials that are currently difficult to create.
- For this study, L-PBF is used due to its superior dimensional accuracy.

High Temperature AM Compatible Materials

High Temperature Materials:

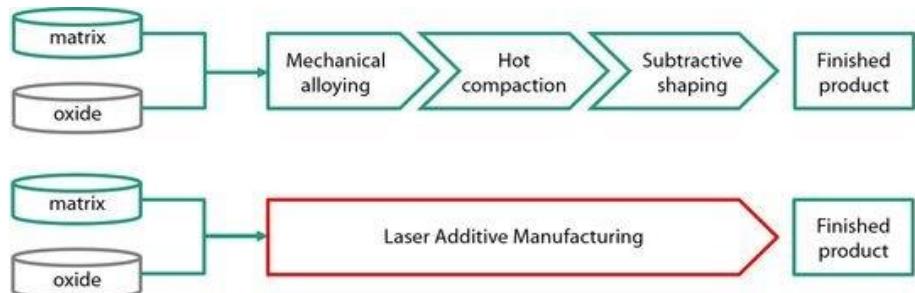
- Refractory metals
- Carbon-Carbon composites
- CMC's
- Ni-base superalloys
- **Oxide Dispersion strengthened (ODS) alloys**



Inspired by Andy Jones. ODS alloy Development.

(ODS) alloys offer higher temperature capabilities compared to Ni-base superalloys. However, it has been a challenge to produce ODS alloys through conventional manufacturing methods.

Conventional Manufacturing vs AM



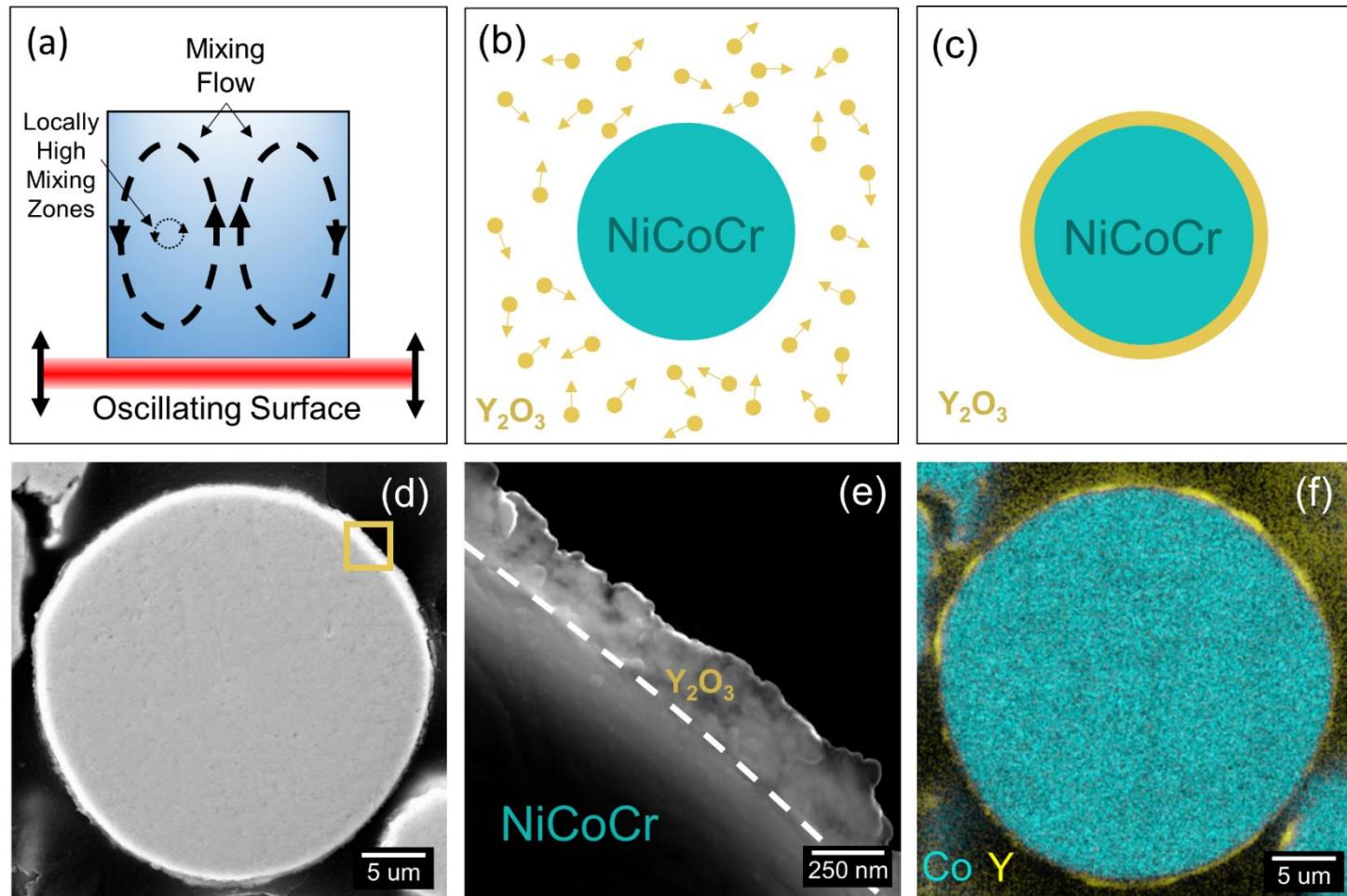
Can AM improve ODS alloy manufacturability?



Methods

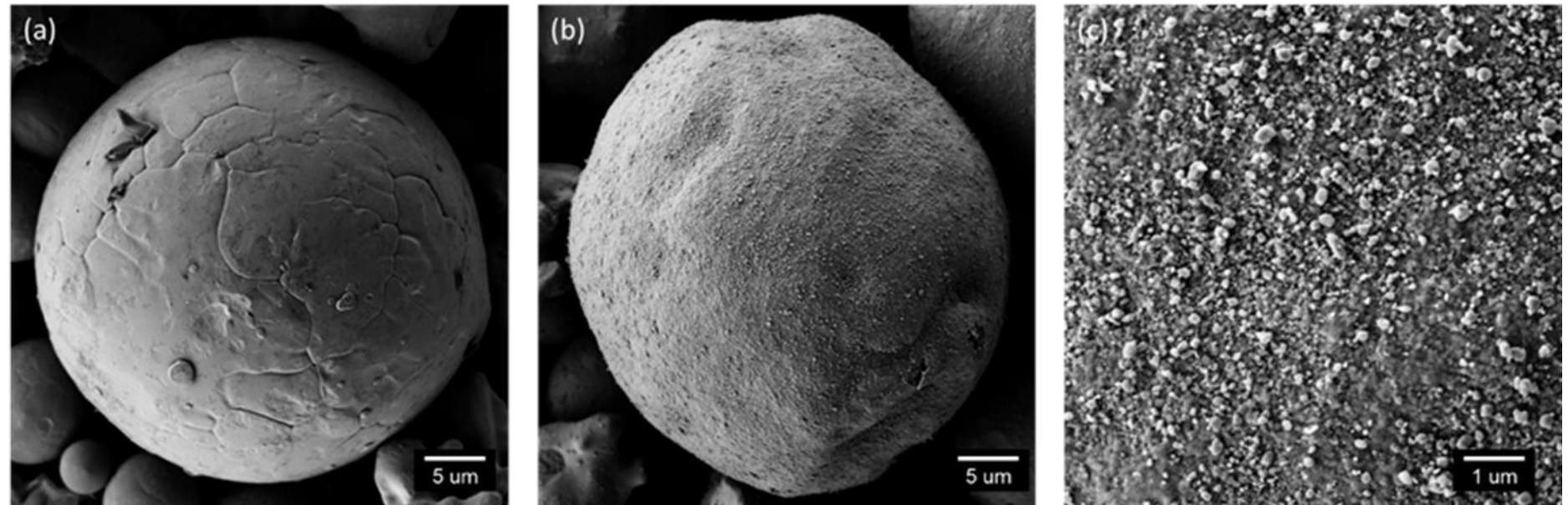
- Micron-scale (10-45um) NiCoCr and NiCoCr-ReB powder was acquired from Praxair.
- Nano-scale (100-200nm) Y_2O_3 powder was acquired from American Elements.
- L-PBF Machine: EOS M100
- Powder Mixing: Resodyn LabRAM II
- Aim of study
 - Leverage L-PBF to produce oxide dispersion strengthened multi-principal element alloys.
 - Determine optimal L-PBF laser parameters for baseline AM NiCoCr, oxide dispersion strengthened ODS-NiCoCr, and ODS-NiCoCrReB (ODS-ReB) builds.
 - Produce 99.9% dense vertical test specimen for microstructural and mechanical analysis using AM NiCoCr, ODS-NiCoCr, and ODS-ReB.
 - Explore heat treatment effects on mechanical performance
 - Produce a high temperature capable 3D printed combustor dome.

LEW-20020-1: “Novel Fabrication Technique for Oxide Dispersion Strengthened (ODS) Alloys”



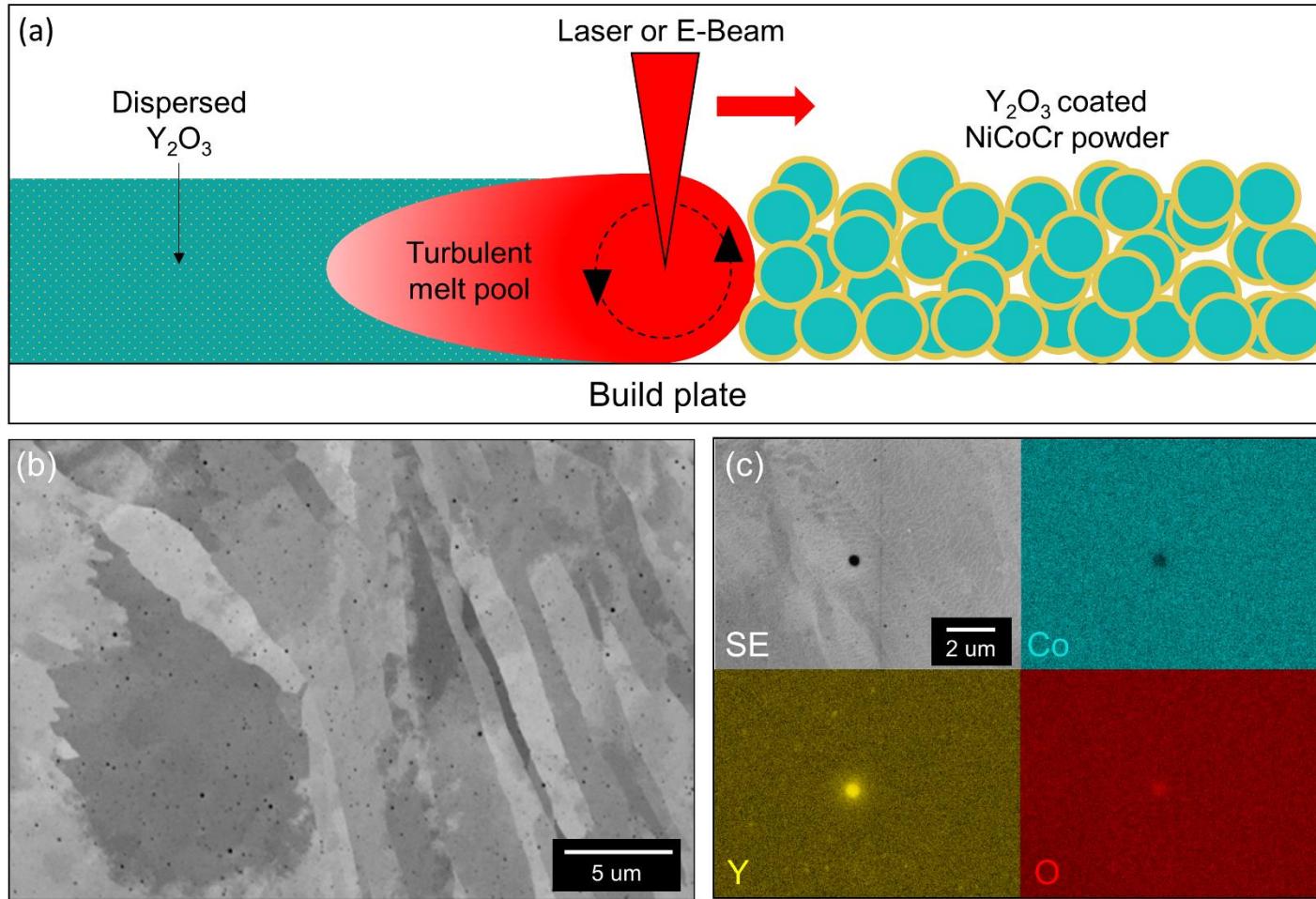
New high energy mixing technique successfully coats NiCoCr powder with 1 wt.% Y_2O_3 .

Novel Powder Coating Technique



- The advanced dispersion coating (ADC) technique did not deform the metallic powder.
- The ADC technique fully coats the metallic powders with nano-scale oxides
- Both uncoated and coated powders qualitatively passed the Hall flow test.
- The technique does not affect the printability of the powder lot.

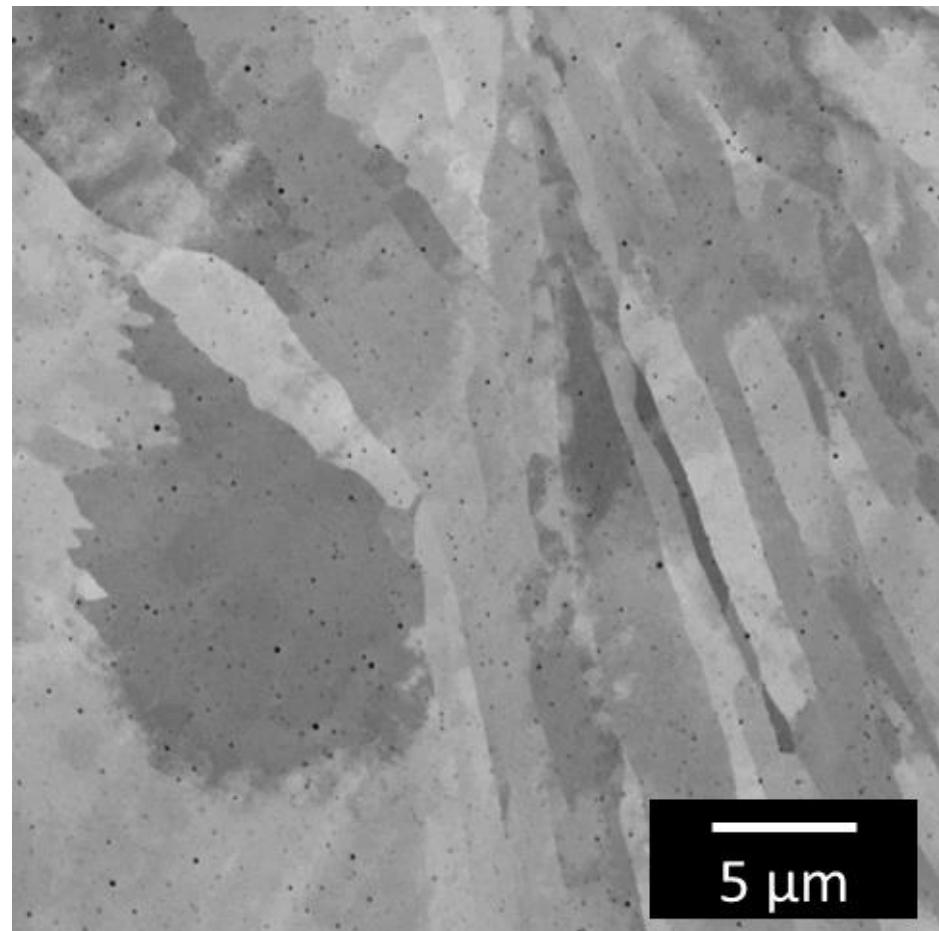
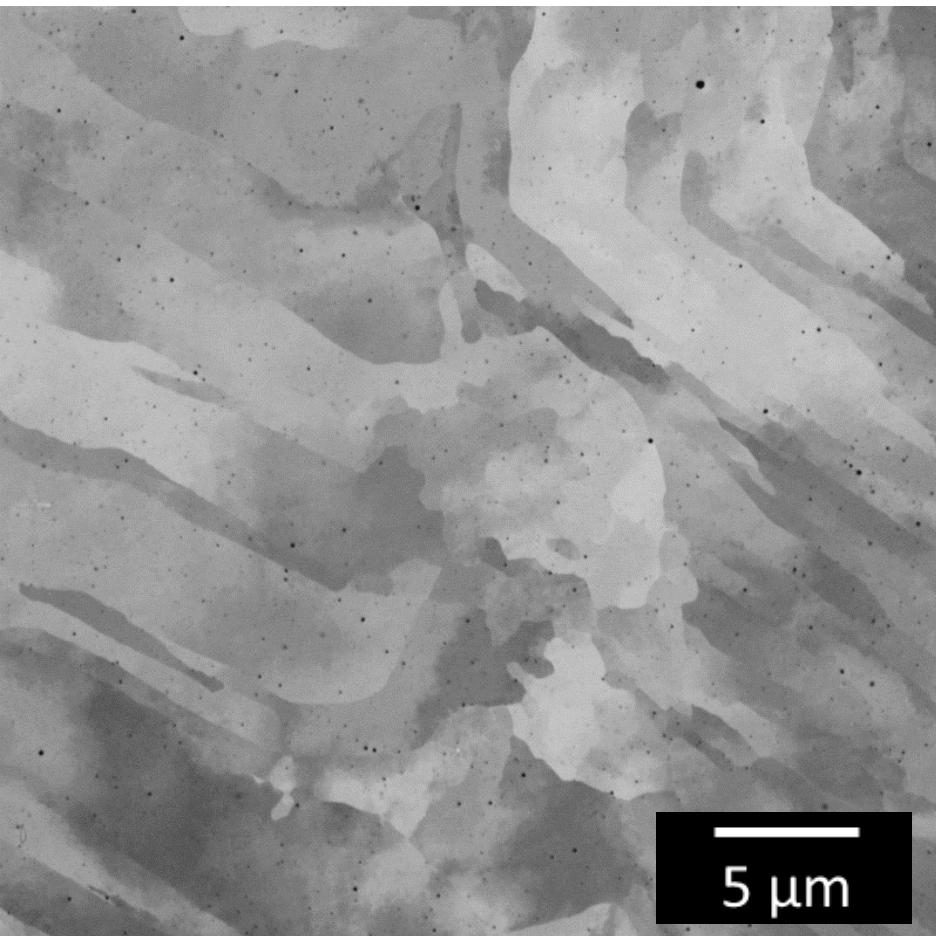
Leveraging L-PBF to Produce Oxide Dispersion Strengthened Alloys



L-PBF successfully disperses the nano-scale Y_2O_3 particles throughout the AM build

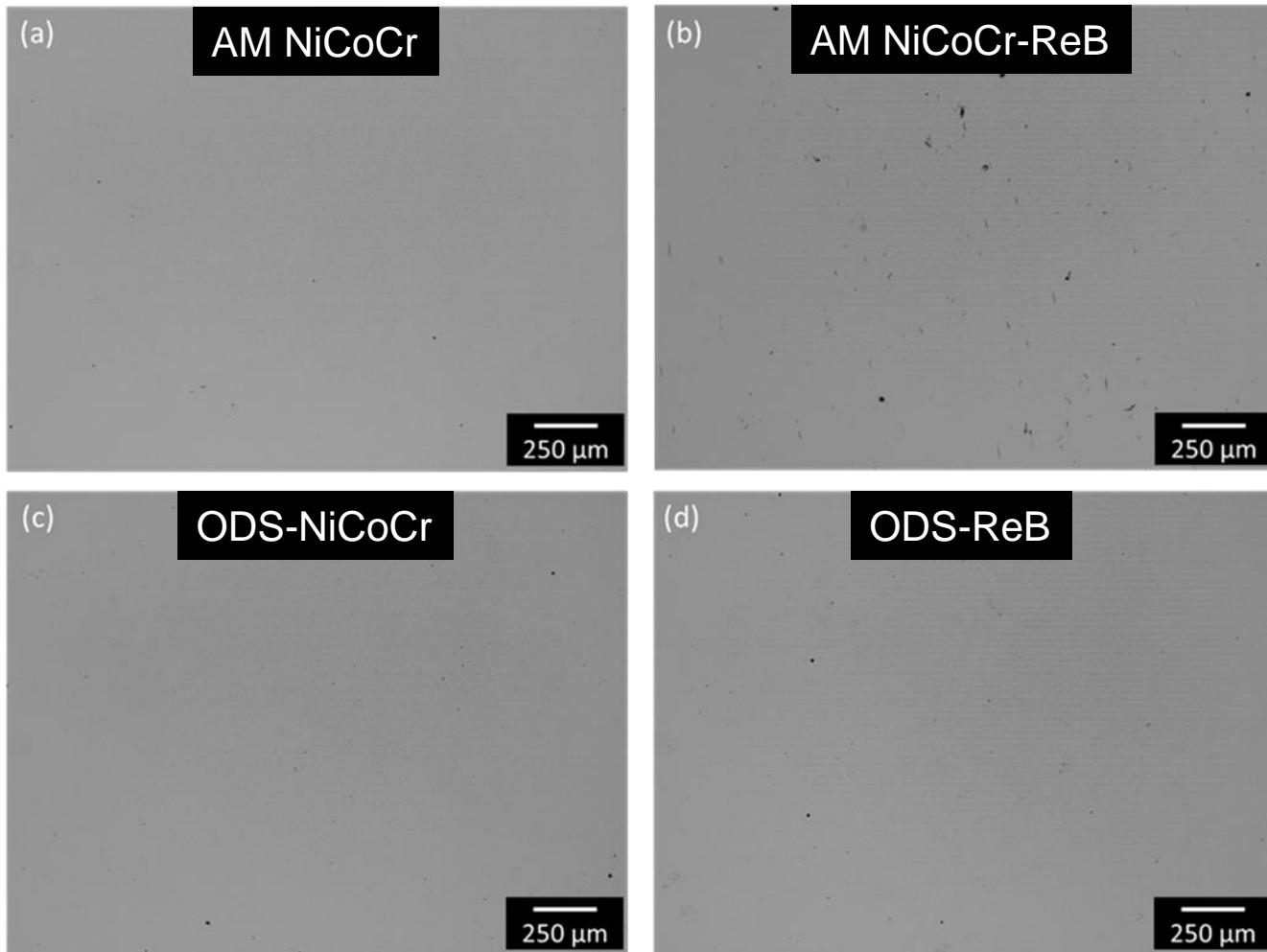


ODS-NiCoCr Microstructure



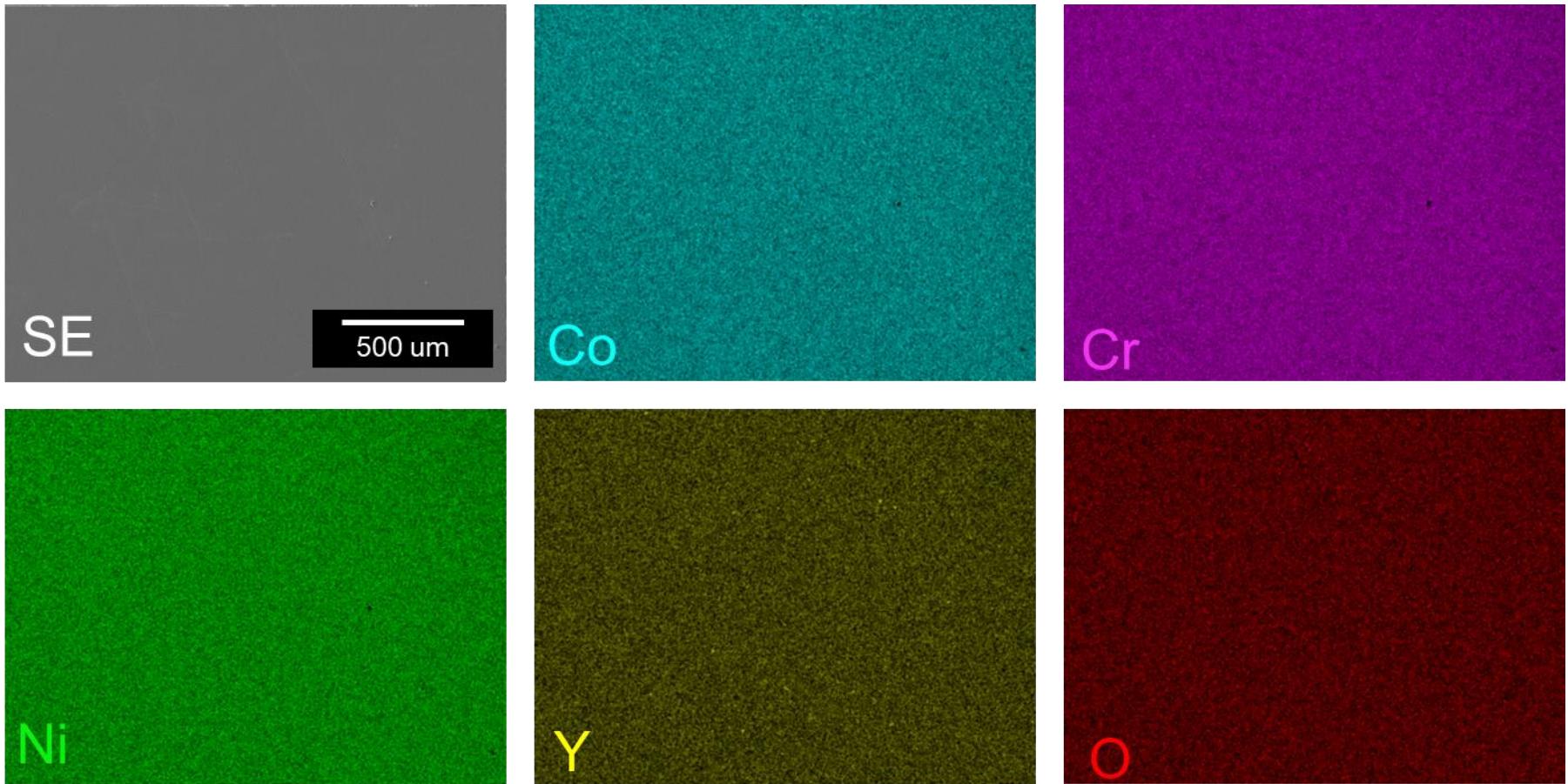
Nano-scale Y_2O_3 particles are randomly dispersed throughout microstructure.

Microstructures - Porosity



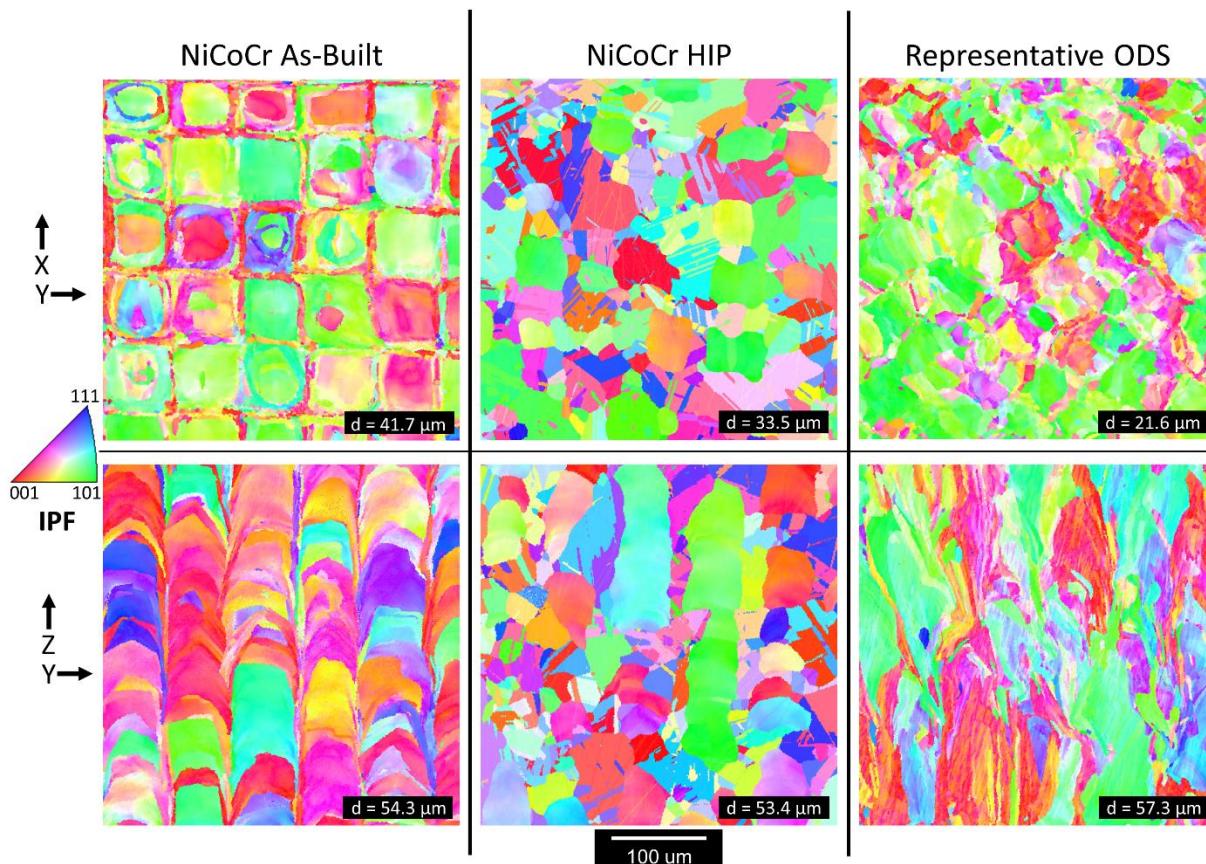
99.9% dense parts were successfully built for AM NiCoCr, ODS-NiCoCr, and ODS-ReB powder lots. Uncoated AM NiCoCr-ReB powder exhibited extensive micro-cracking. Result suggests coated powder is more printable.

EDS – NiCoCr-ODS Microstructure



- Large ($>20\text{um}$) Y_2O_3 particles are not present in AM builds
- NiCoCr matrix remained a random solid solution during L-PBF process.

Microstructure Analysis



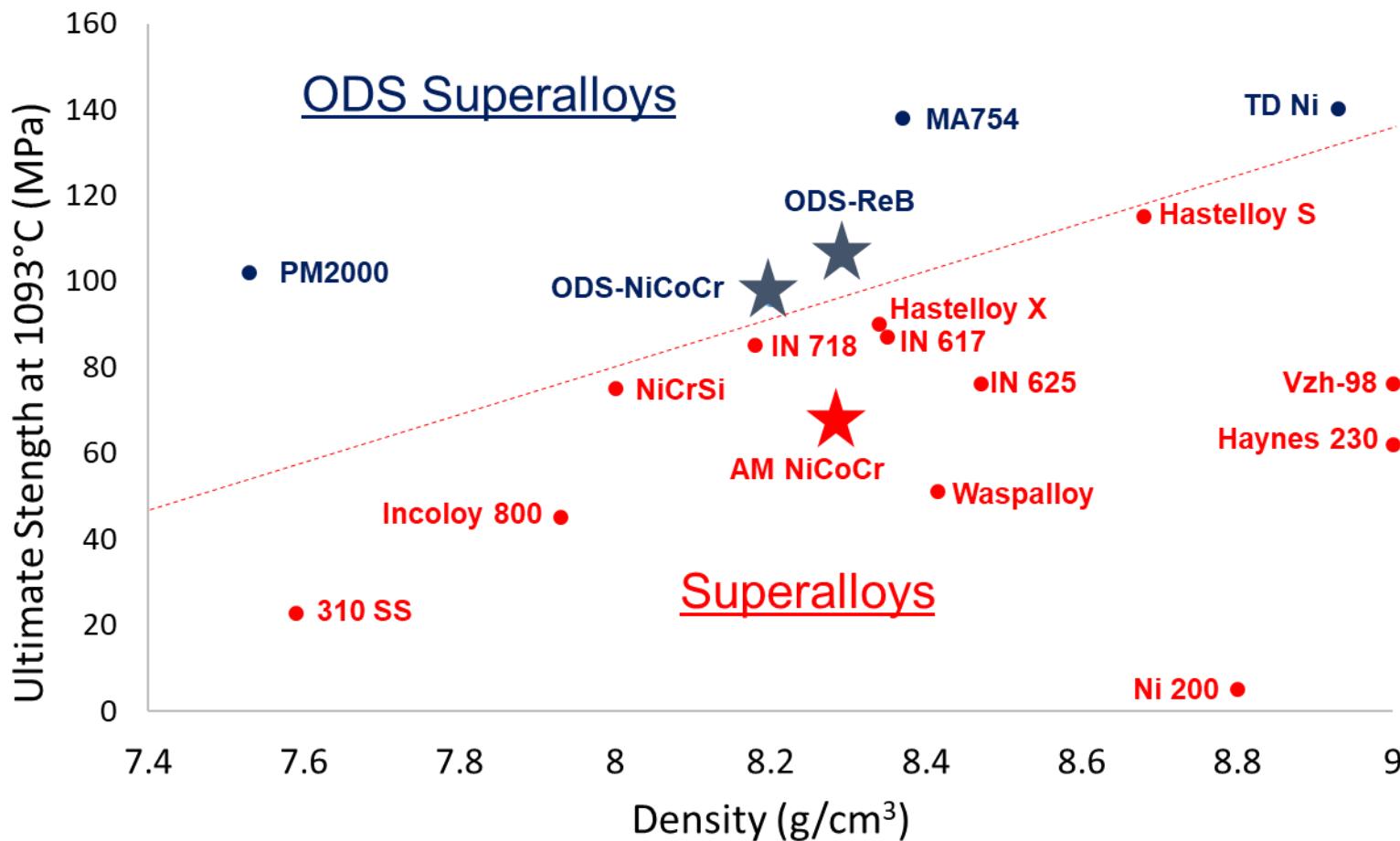
- Y_2O_3 particles have pinned the grain boundaries in the ODS AM builds.
- The HIP cycle successfully removed residual stresses for AM samples

Residual Stress

Alloy	As-Built – Build direction	As-Built – 90° from build direction	HIP – Build direction	HIP – 90° from Built direction
AM NiCoCr	34 ± 35	141 ± 96	-5 ± 3	-4 ± 6
ODS-NiCoCr	320 ± 51	185 ± 49	-11 ± 9	-12 ± 9
ODS-ReB	321 ± 52	179 ± 47	-8 ± 5	-12 ± 6



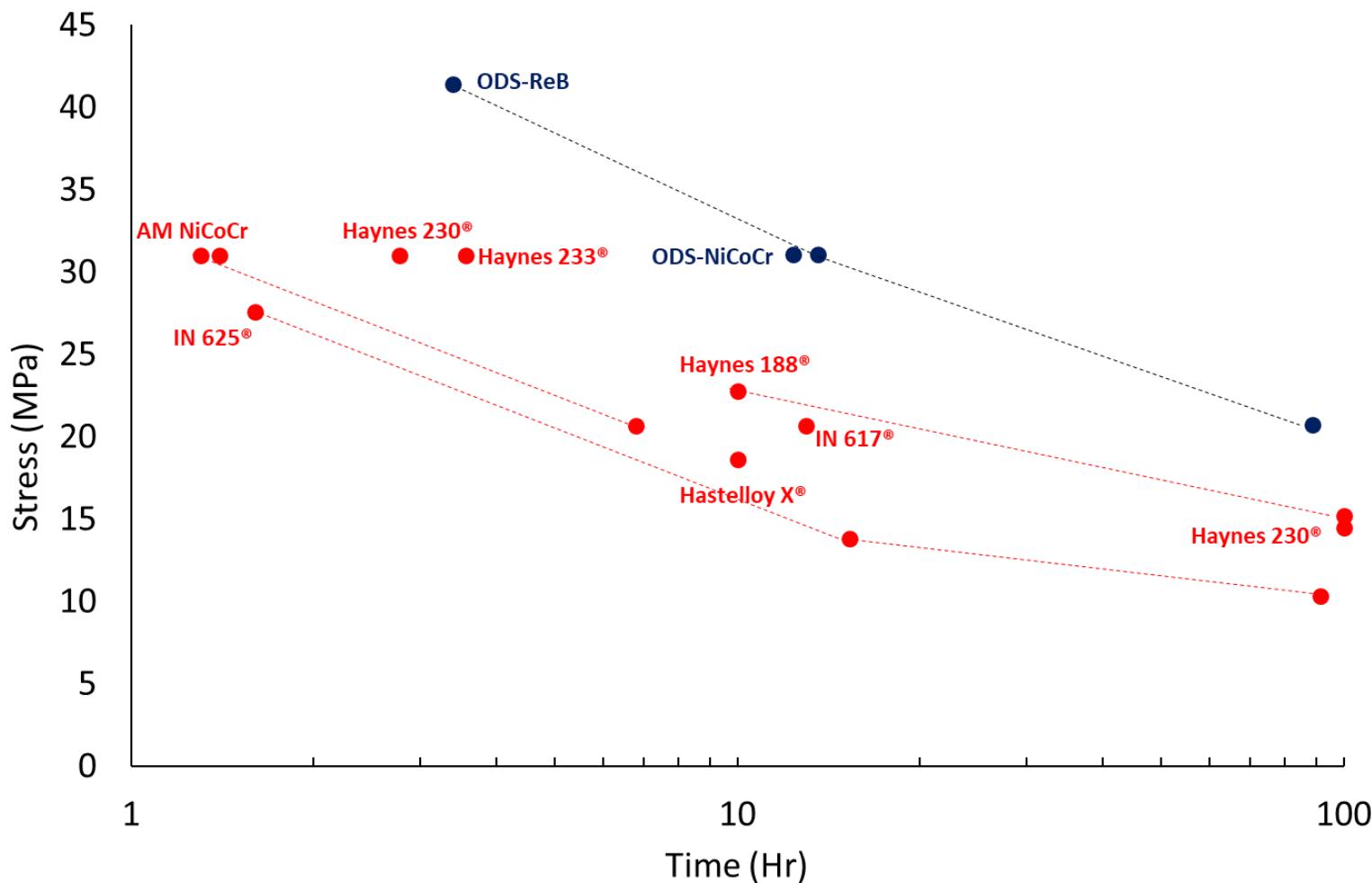
Tensile Strength vs Density Comparison



Scatter plot confirms the successful production of a ODS alloy using AM



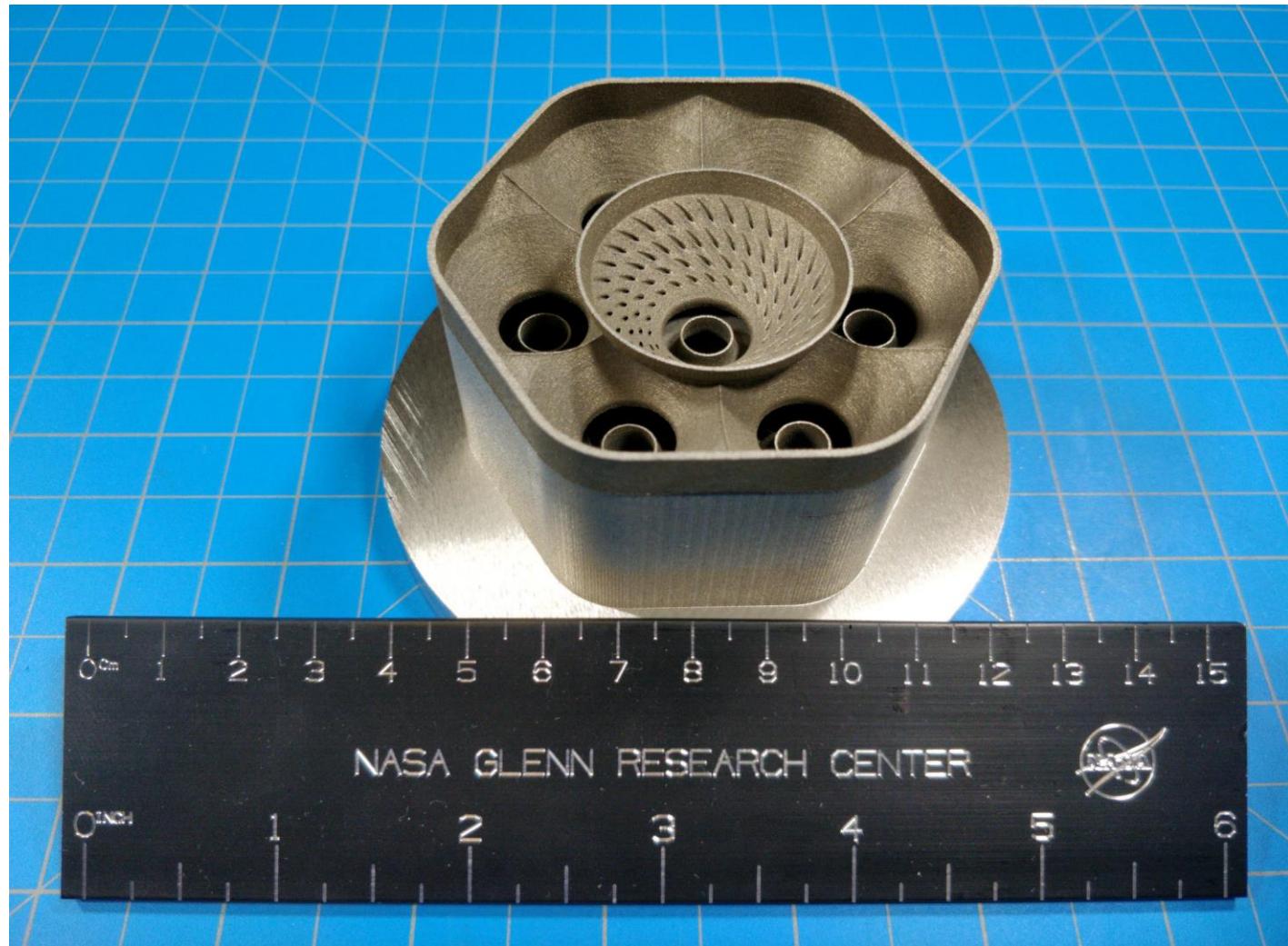
Creep Rupture Lives Comparison- 1093°C



Scatter plot confirms the successful production of a ODS alloy using AM



Oxide Dispersion Strengthened MPEA Combustor Dome





LEW-19886-1: “Additively Manufactured Oxide Dispersion Strengthened Medium Entropy Alloys for High Temperature Applications”

Model Driven MPEA Design

Goals to improve on previous NiCoCr Entropy Alloy:

- 1.) Maximize solid solution strengthening
- 2.) Maintain solid solution matrix
- 3.) Add grain boundary carbides
- 4.) Reduce freezing range to under 100°C for printability

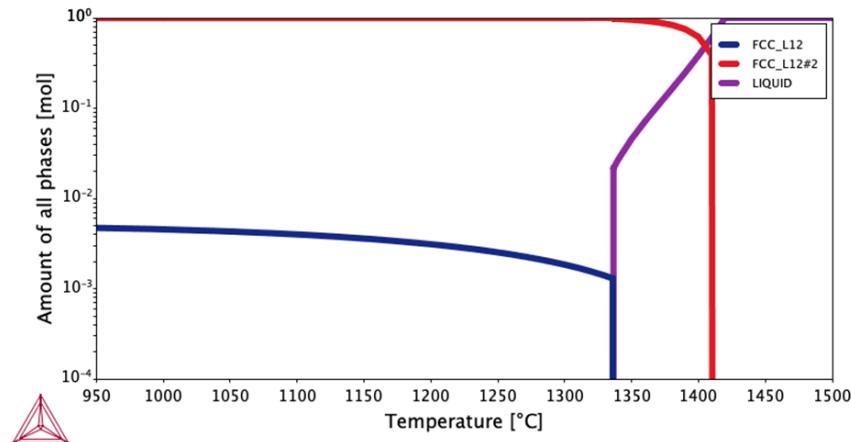


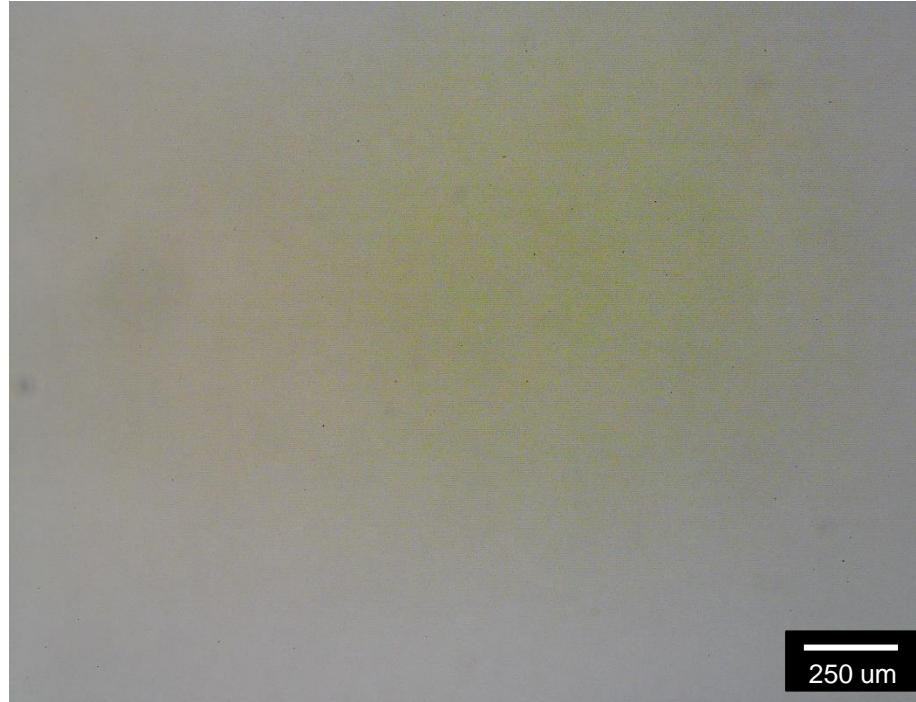
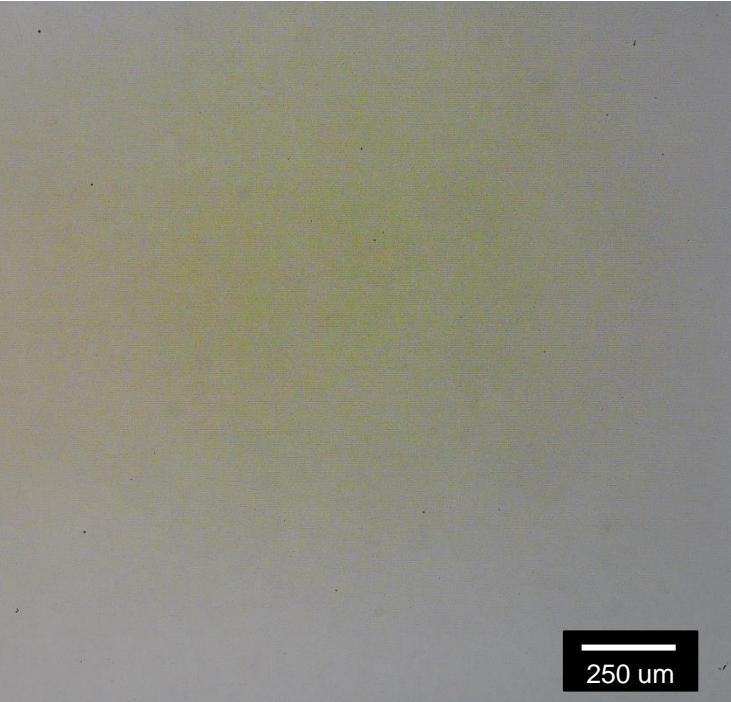
Figure: CALPHAD simulation of phase formation in new composition. No intermetallic or TCP phases are predicted.

Over 50 simulations provided an optimized composition named Alloy X

Models calculated by C. Kantzos



Optical Microscopy – Alloy X - ODS HIPed



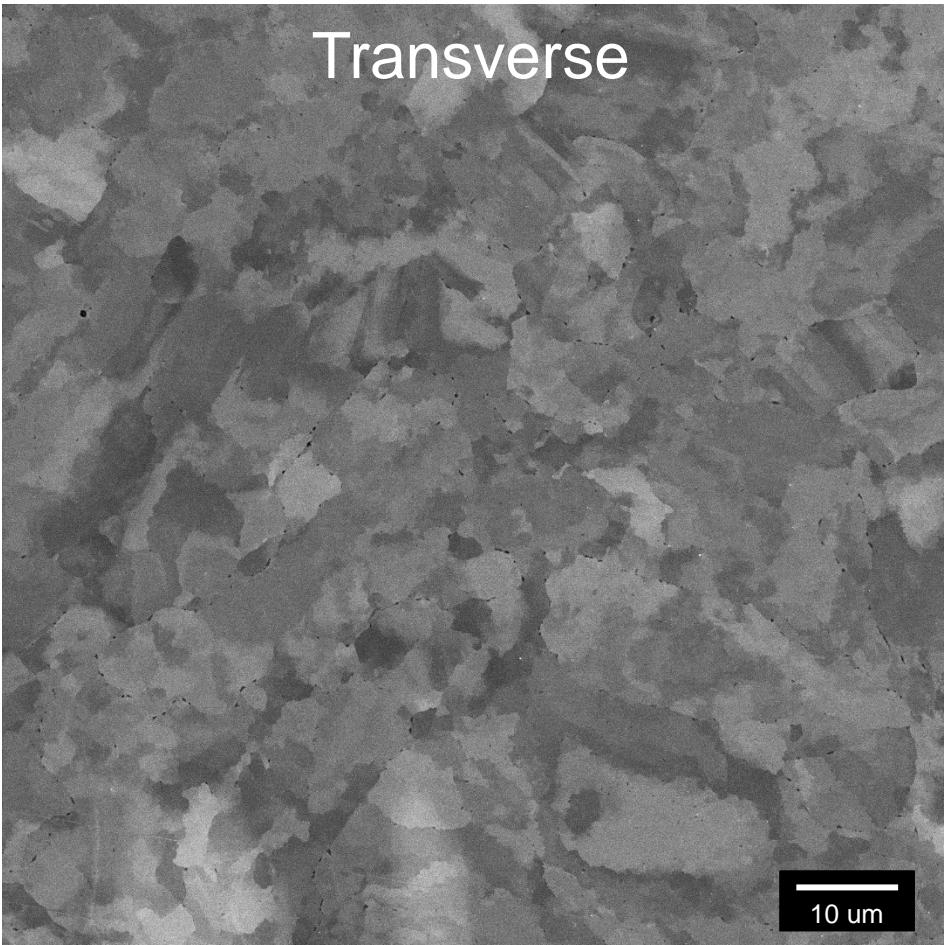
HIP removes most defects.

Average 99.9912% density

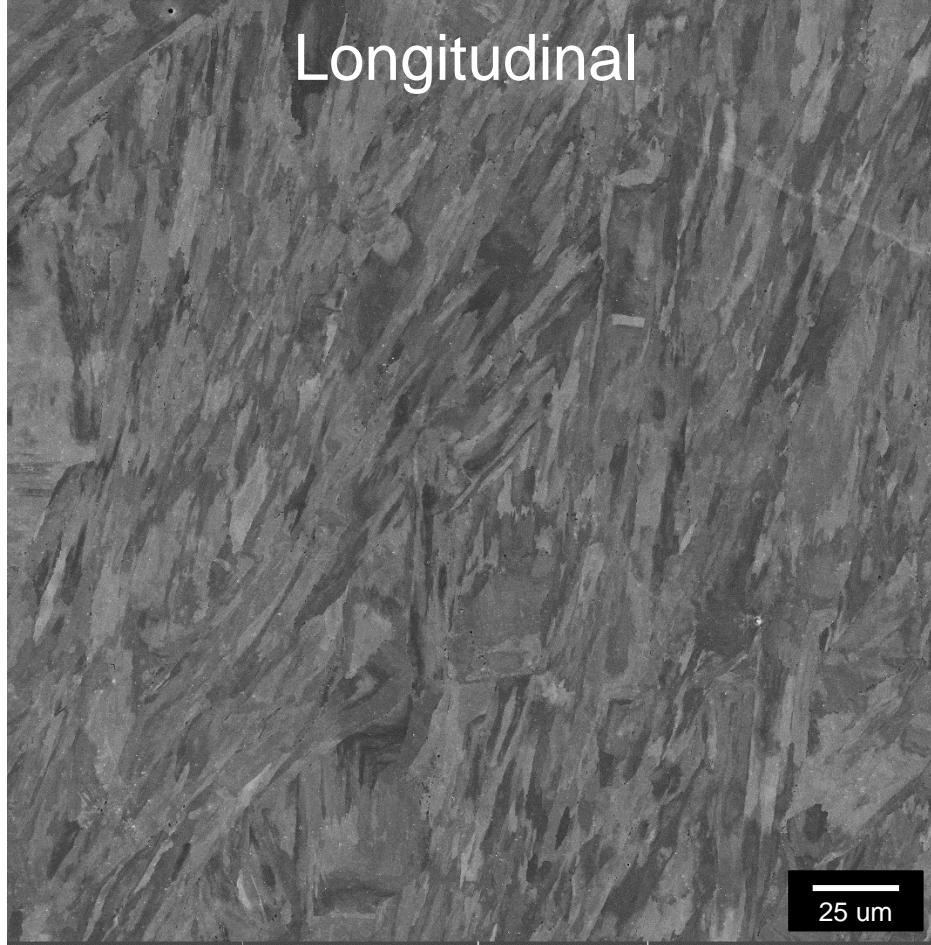


SEM – Alloy X - ODS HIPed

Transverse



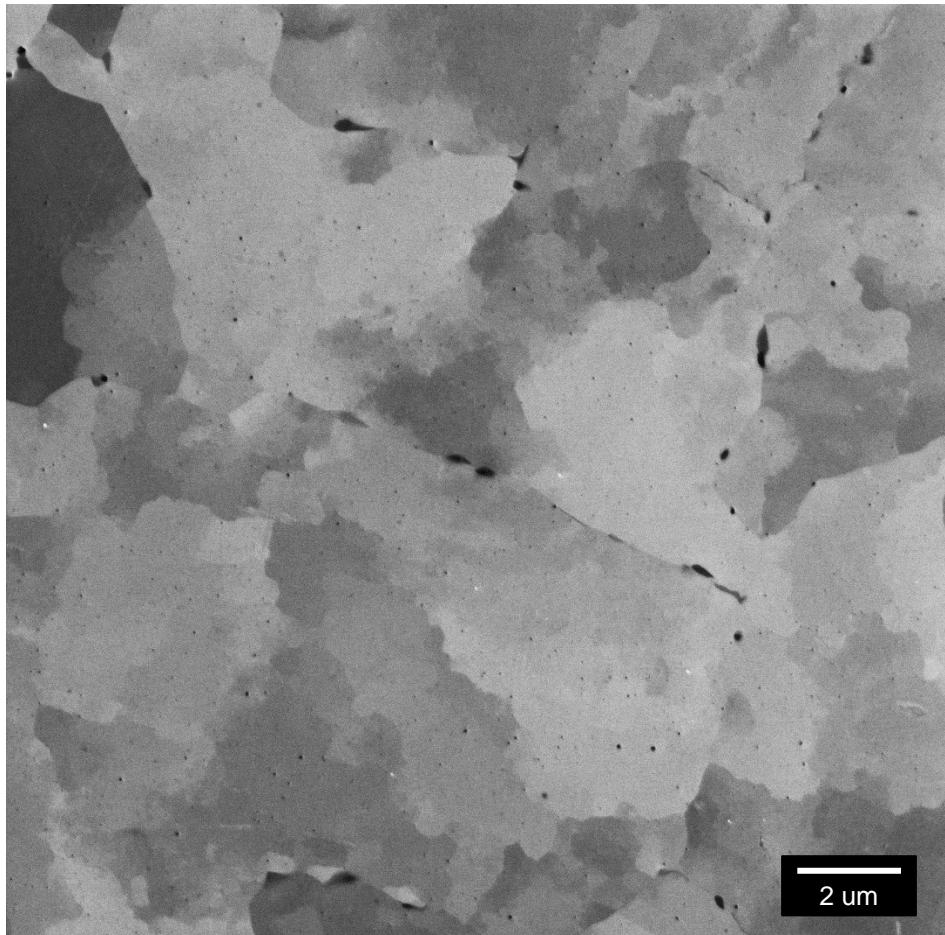
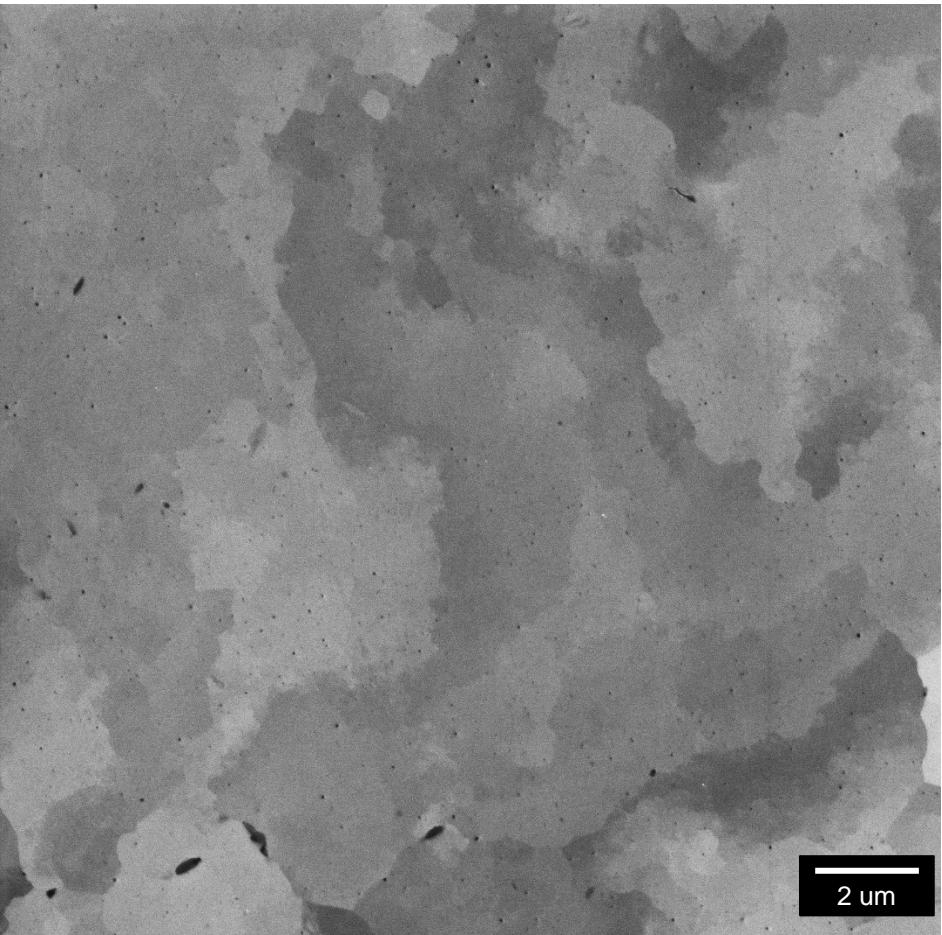
Longitudinal



Low magnification SEM images reveal anisotropic grain structure. Suggests oxides are present pinning grain boundaries during HIP

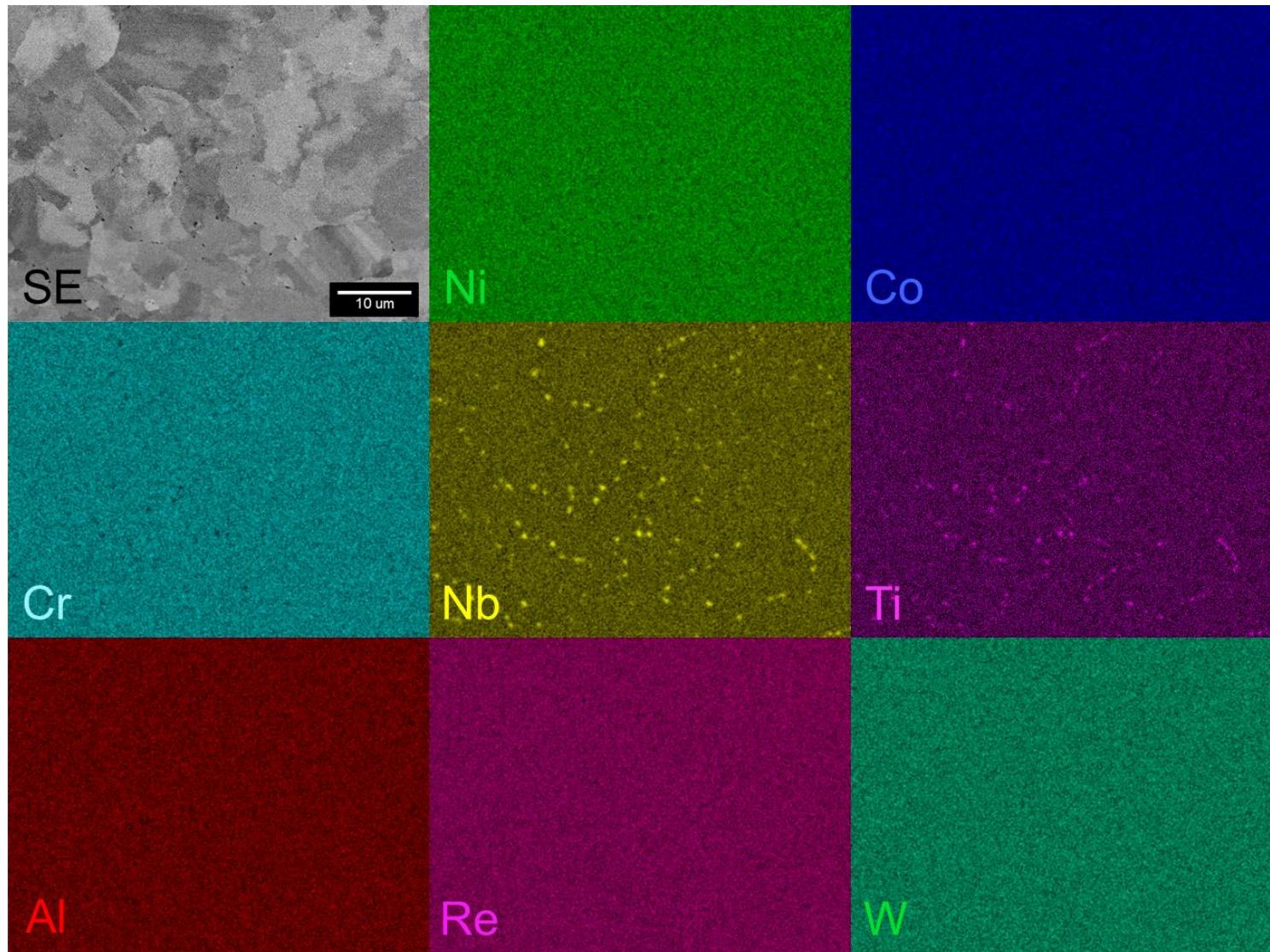


SEM – Alloy X - ODS HIPed

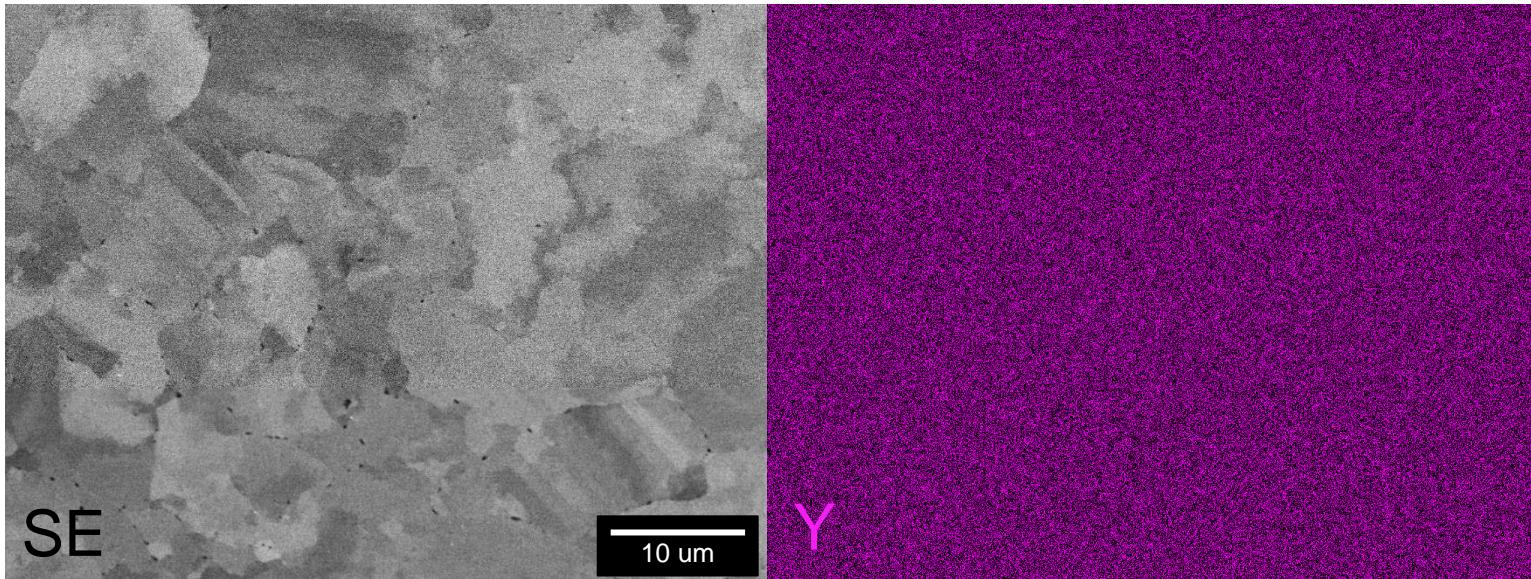


High resolution SEM reveals dispersed nano-oxides and grain boundary phases.

SEM-EDS – Alloy X - ODS HIPed



SEM-EDS – Alloy X - ODS HIPed

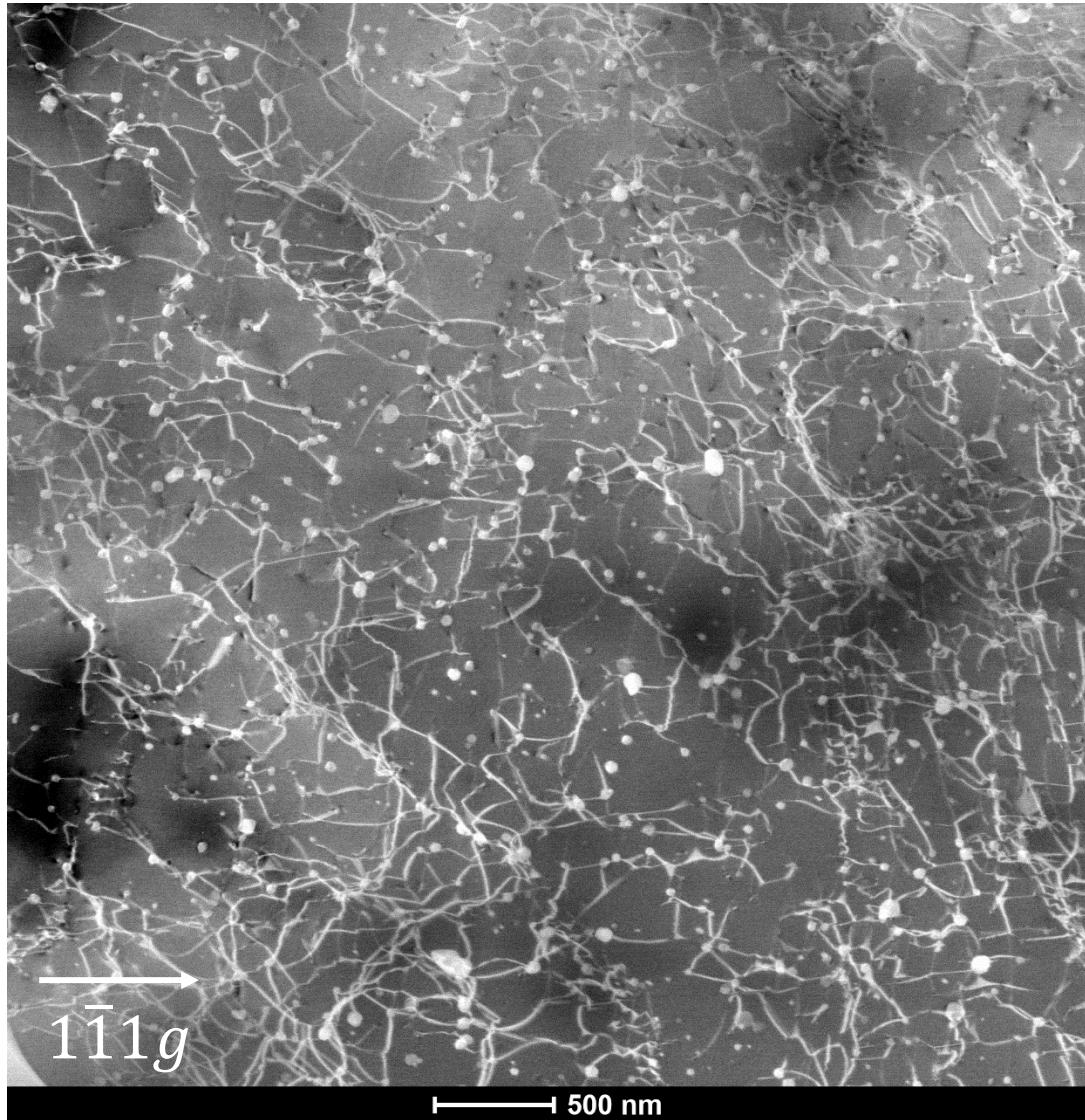


- SEM-EDS confirms the presence of Nb- and Ti-rich carbides along grain boundaries.
- SEM-EDS confirms the new composition has maintained a solid solution matrix.
- No intermetallic or TCP phases were observed.
- Bulk Y_2O_3 appears to have been avoided as well.

Confirmation of CALPHAD model predictions

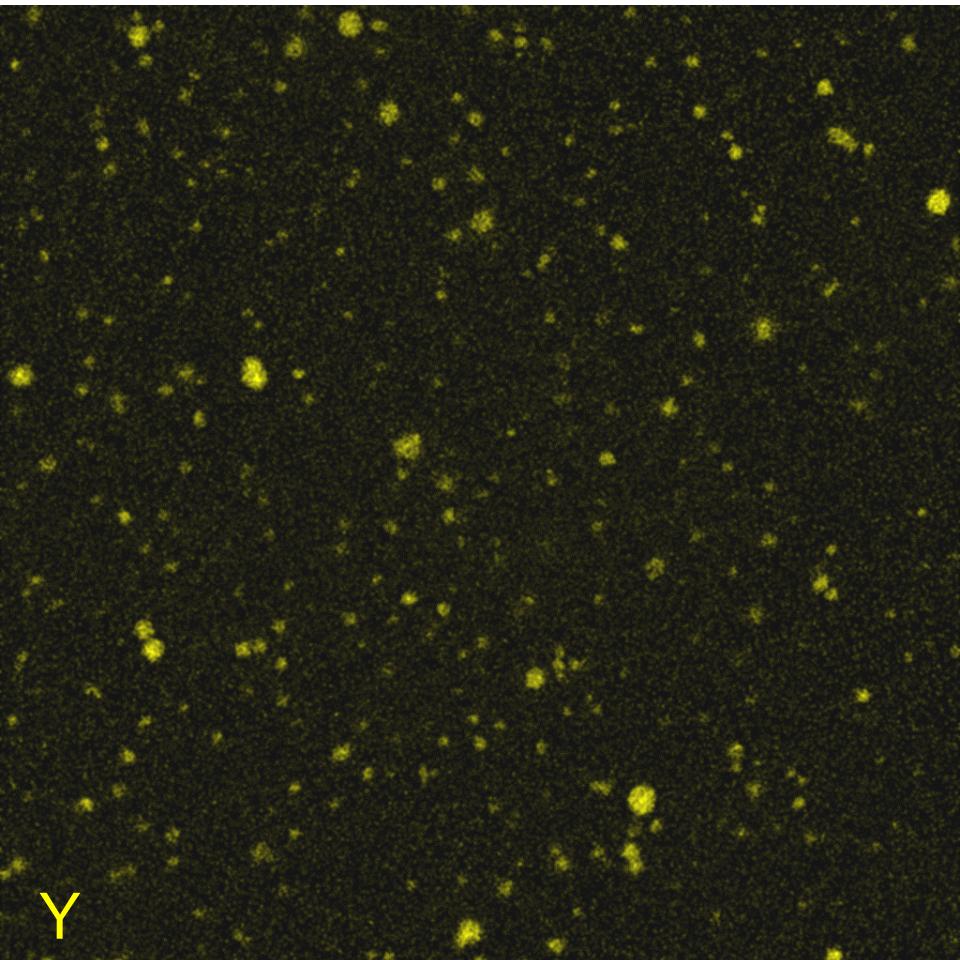
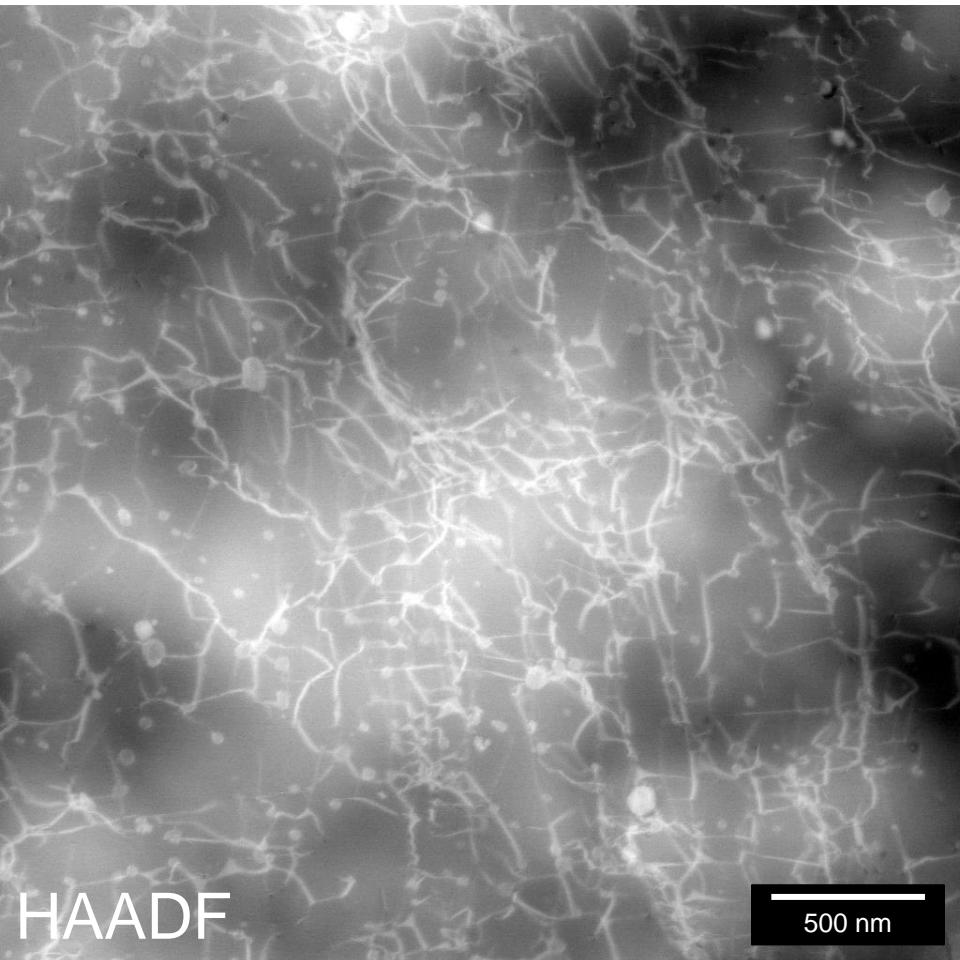


STEM-EDS Analysis



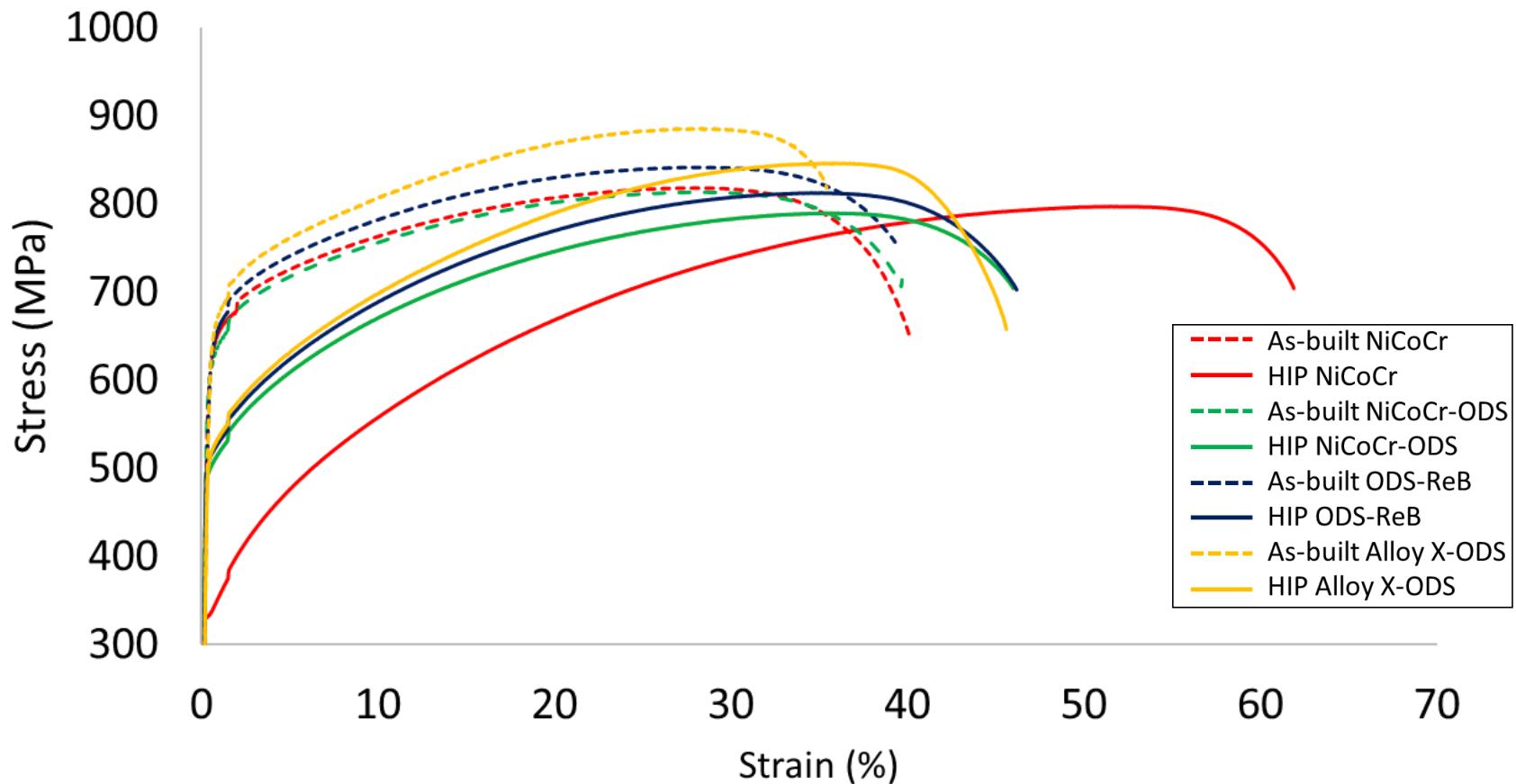
HAADF STEM analysis confirms presence of nano dispersoids within Alloy X ODS matrix. Highly dense dislocation networks still present after HIP step due to dislocation pinning by dispersoids.

STEM-EDS



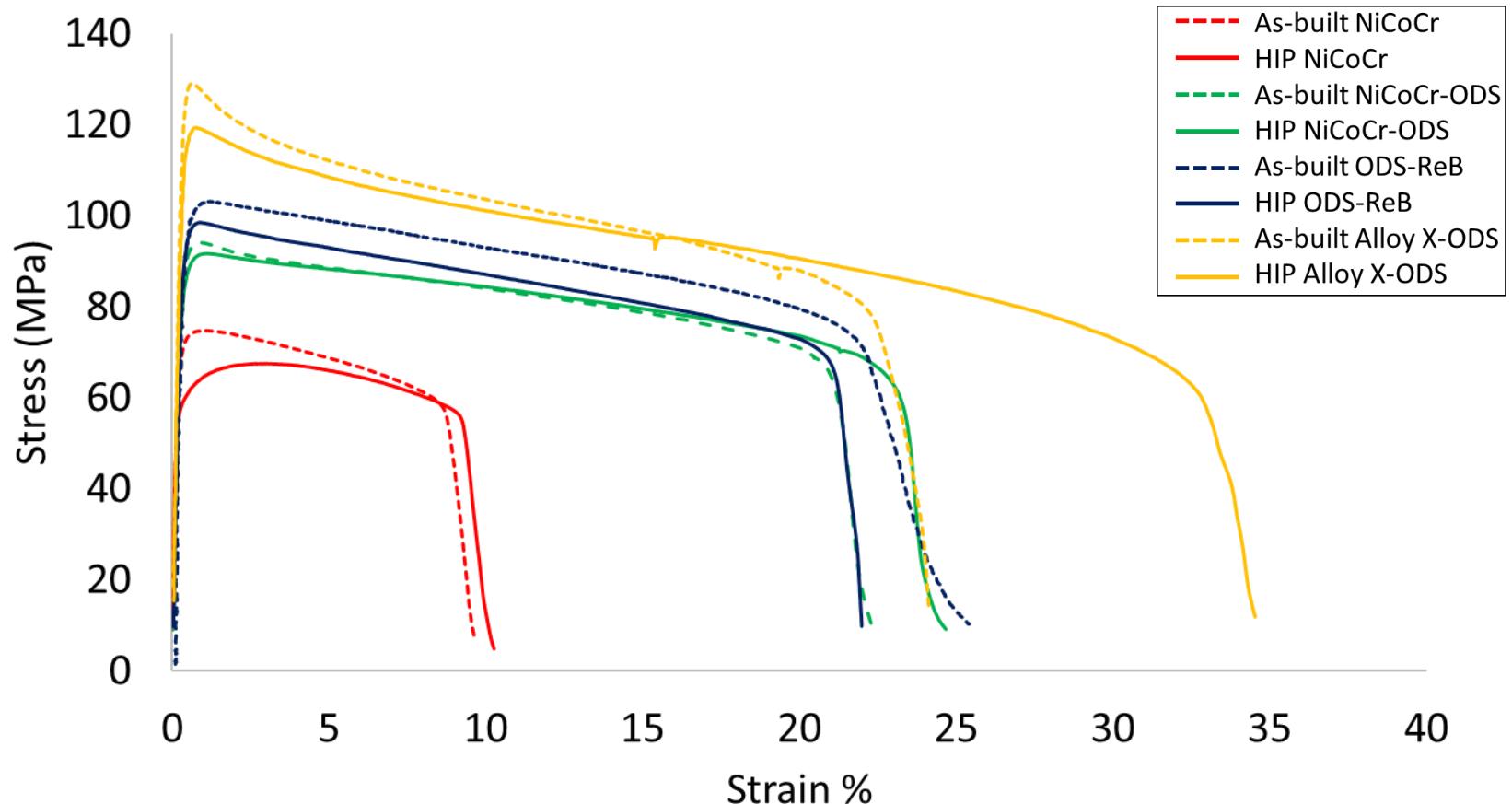
STEM-EDS analysis confirms high density of nano-scale Y_2O_3 particles throughout bulk. No oxide agglomeration was present. Most other elements did not react with oxides (Nb and Ti were exceptions).

Mechanical Results – Room Temperature Tensile



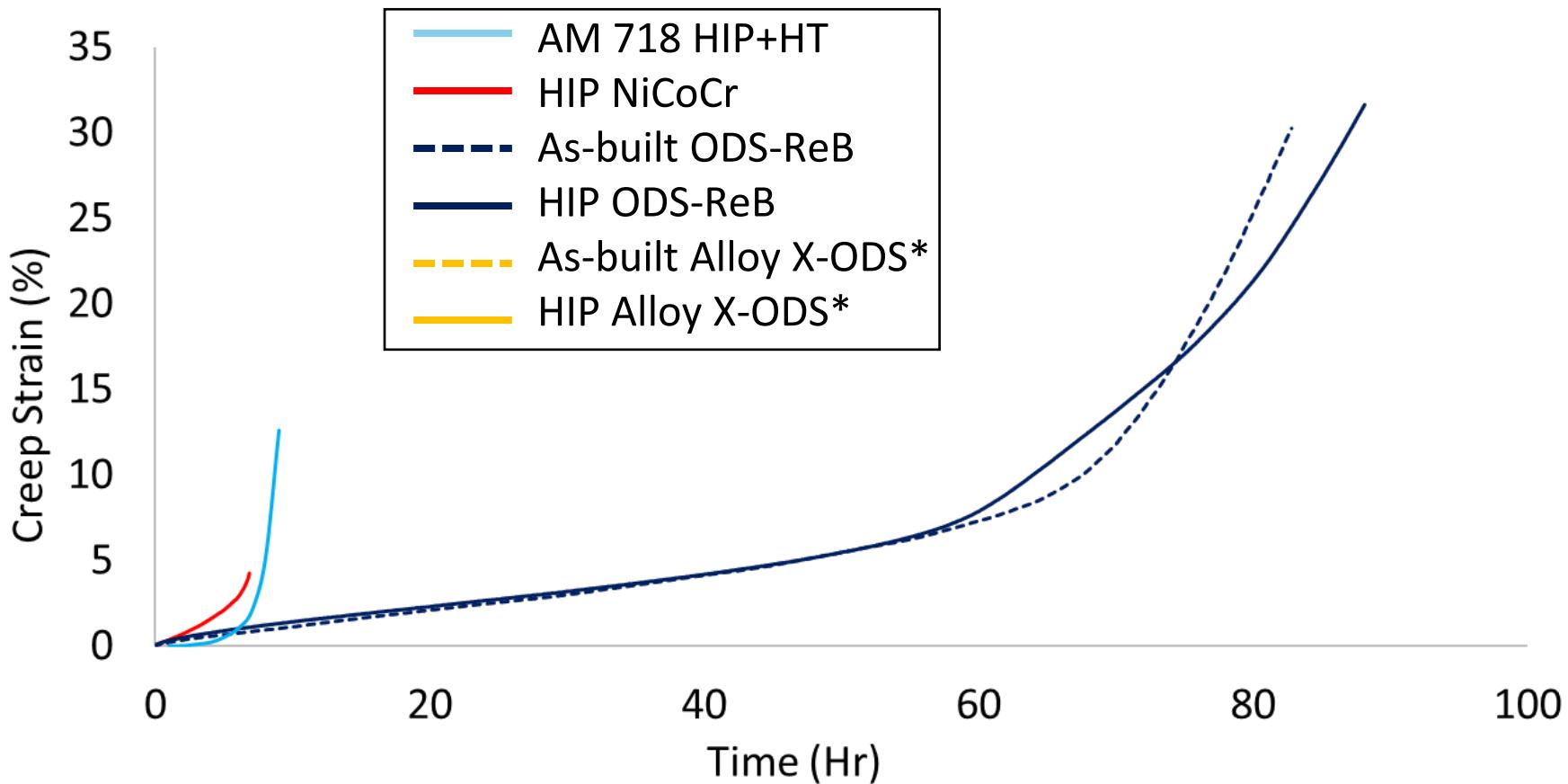
- X-ODS presents higher ultimate tensile strength at room temperature while maintaining the MPEAs fundamental high ductility.

Mechanical Results – 1093°C Tensile



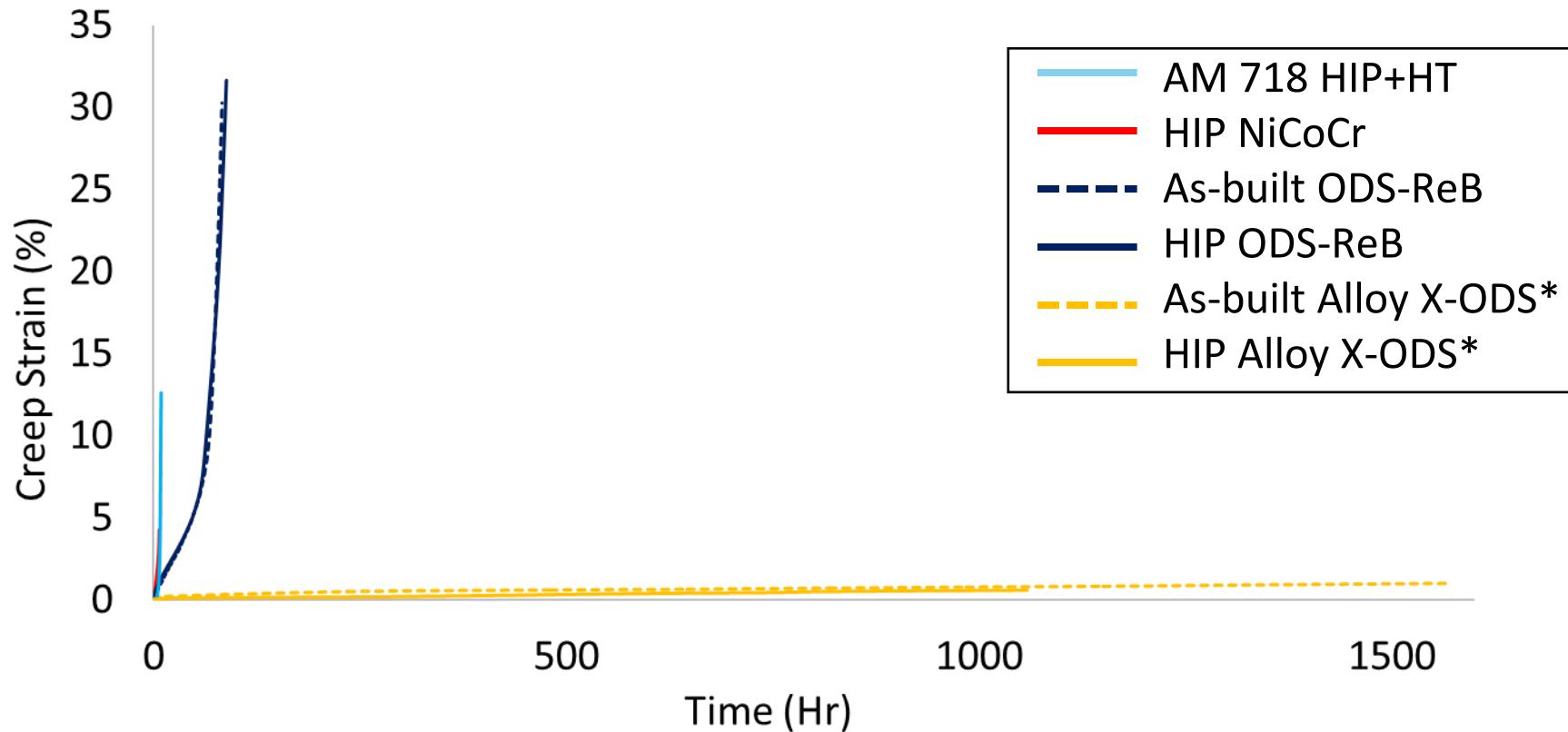
- X-ODS provides almost double the tensile strength and over 3x the ductility compared to AM NiCoCr.

Mechanical Results – 1093°C/20MPa Creep Rupture



Incorporating oxides provides significant improvement in creep rupture life at 1093°C

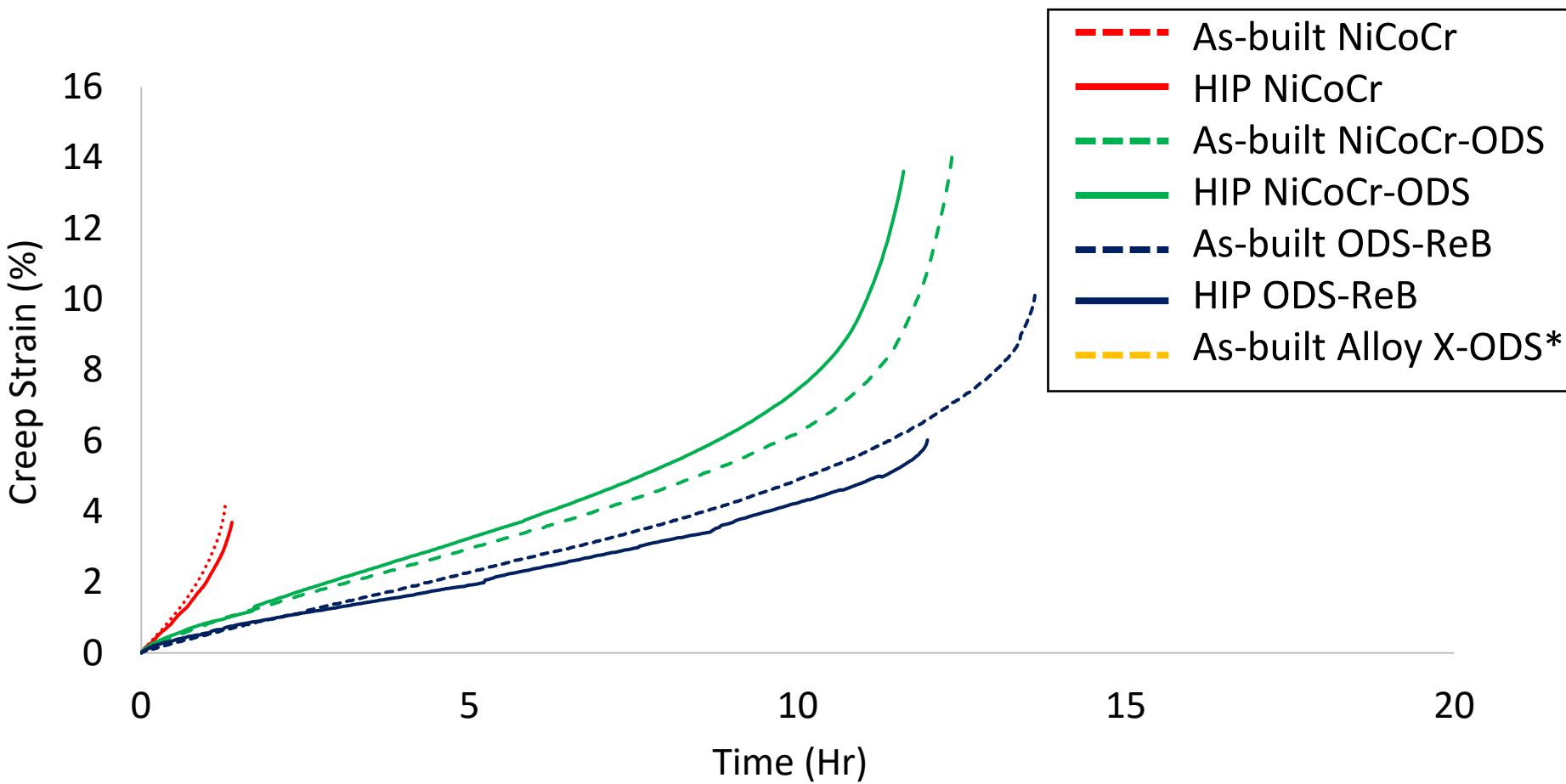
Mechanical Results – 1093°C/20MPa Creep Rupture



- *The as-built Alloy-X ODS test is still running ATM (1% strain at 1566 Hr).
- X-ODS is showing over a 225x improvement over NiCoCr in creep rupture life and almost a 75x improvement over superalloy 718.
- AM 718 built and tested by NASA's Henry DeGroh and Chris Kartzos

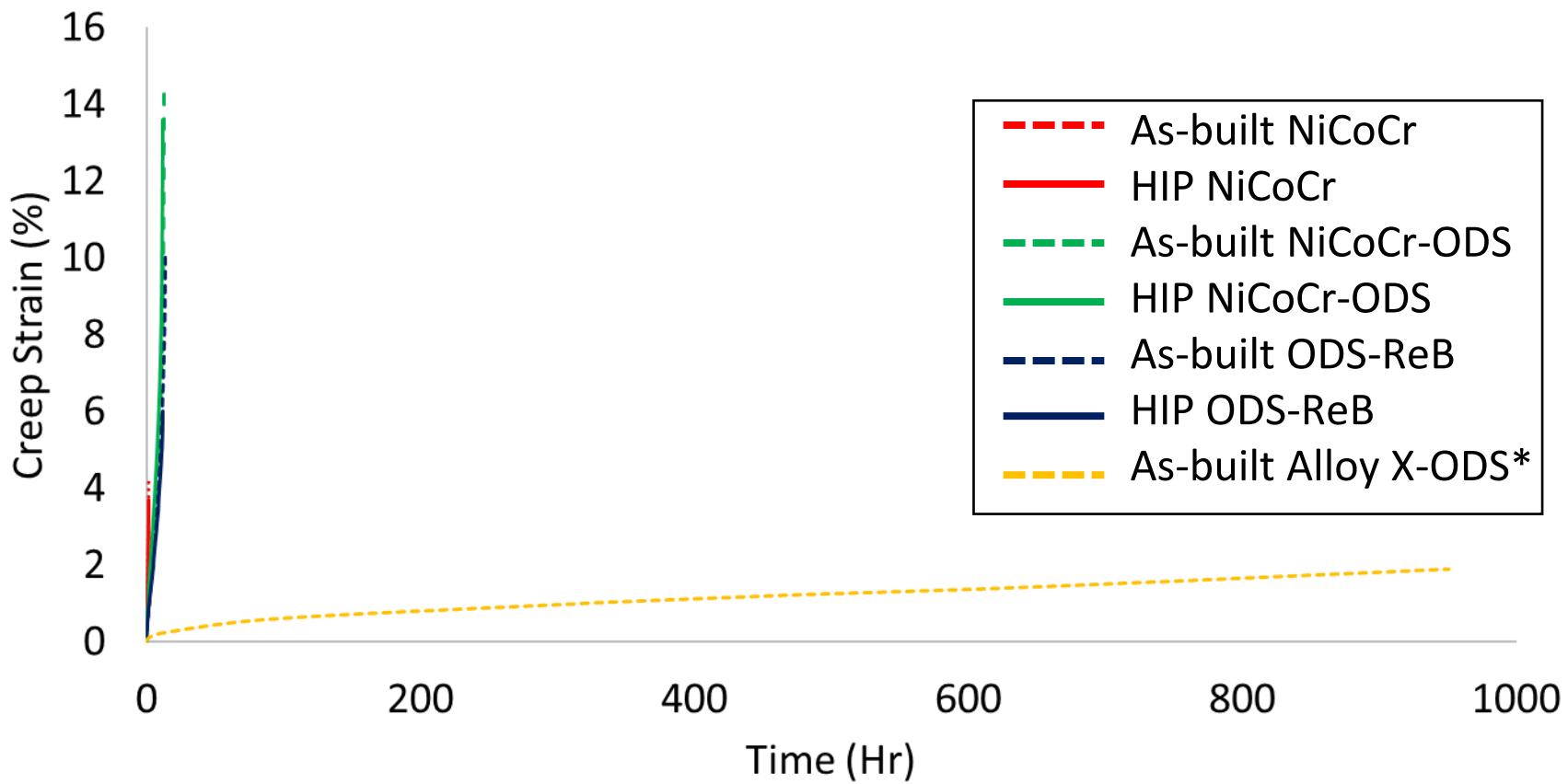
Mechanical Results – 1093°C/31MPa

Creep Rupture



Incorporating oxides provides significant improvement in creep rupture life at 1093°C

Mechanical Results – 1093°C/31MPa Creep Rupture

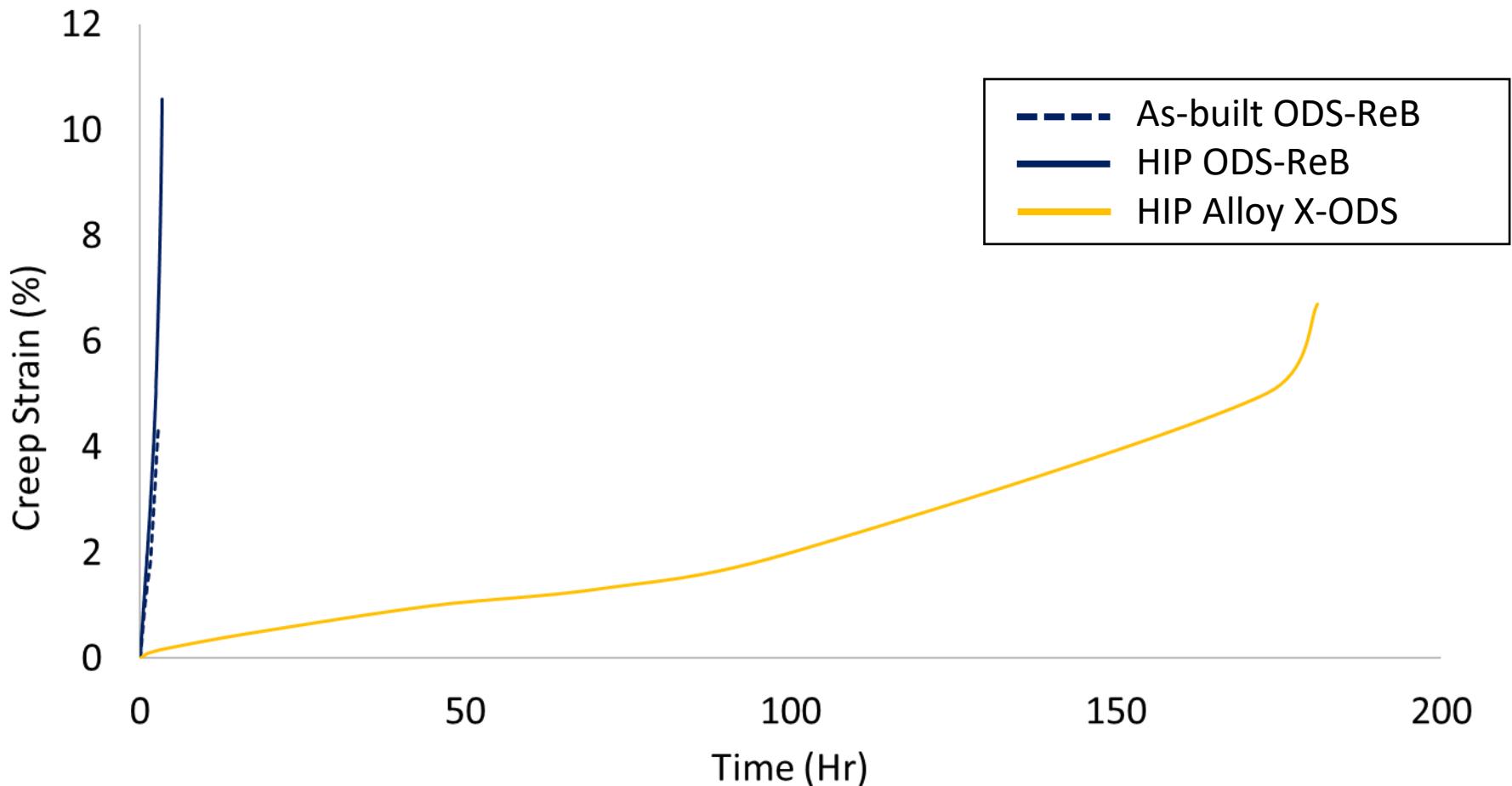


- *The as-built Alloy-X ODS test is still running ATM
- X-ODS is showing over a 750x improvement over NiCoCr in creep rupture life.



Mechanical Results – 1093°C/41MPa

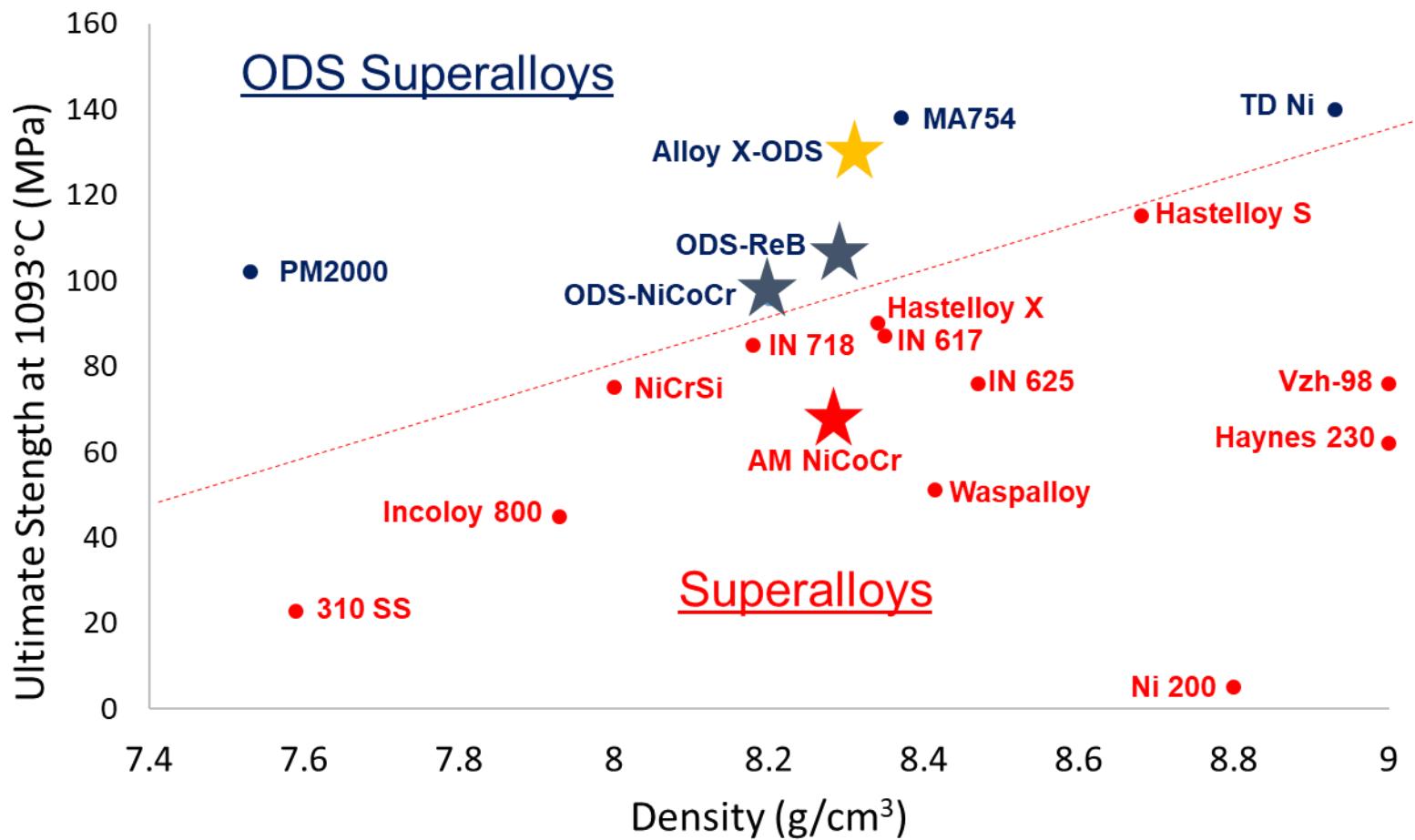
Creep Rupture



X-ODS survived 50x longer than HIPed ODS-ReB during creep at this stress and temperature.



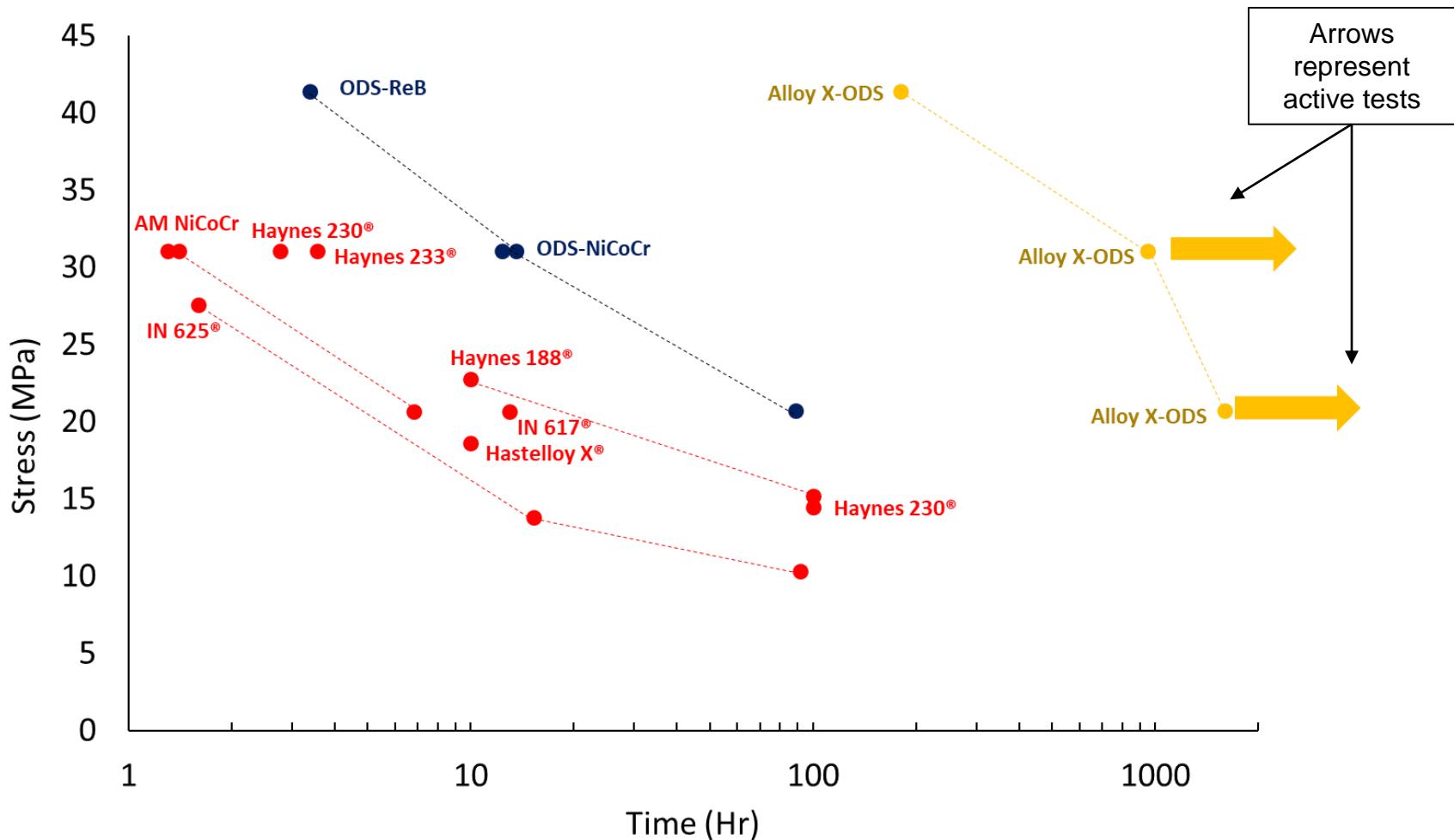
Tensile Strength vs Density Comparison



Scatter plot confirms the successful production of a ODS alloy using AM



Creep Rupture Lives Comparison- 1093°C



Even using the current unfinished creep data – Alloy X-ODS is revealing creep properties far superior to conventional superalloys at 1093°C.



Conclusions

LEW-20020-1: “Novel Fabrication Technique for Oxide Dispersion Strengthened (ODS) Alloys

- AM can be leveraged to economically produce oxide dispersion strengthened alloys that until now had been cost prohibitive.
- The advanced dispersion coating technique can successfully coat metallic powders with nano-scale ceramics.
- We believe this new manufacturing technique combined with MPEAs opens a new alloy design space for future high temperature alloys

LEW-19886-1: “Additively Manufactured Oxide Dispersion Strengthened Medium Entropy Alloys for High Temperature Applications”

- Thermodynamic models correctly predicted a stable solid solution matrix phase for Alloy X.
- SEM and TEM characterization confirms a uniform dispersion of nano-scale oxides throughout the alloy X – ODS build
- High temperature mechanical testing of Alloy X – ODS reveals surprising and superior results compared to previous ODS alloys produced within this project and conventionally manufactured high temperature alloys.



Acknowledgments

Questions?



- ASG
- Dave Ellis
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- Joy Buehler
- Bob Carter
- Pete Bonacuse
- Chris Kantzos
- Cheryl Bowman
- Tim Gabb



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